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Air, Water, and Food

FROM A SANITARY STANDPOINT

BY

ALPHEUS G. WOODMAN AND JOHN F. NORTON

Associate Professor of Food Analysis

Assistant Professor of Chemistry of Sanitation

MASSACHUSETTS INSTITUTE OF TECHNOLOGY

"These cannot be taken as sufficient . . . in these times when every word spoken finds at once a ready doubter, if not an opponent. They are, however, specimens, and will serve to make comparisons in time to come." — ANGUS SMITH.

"The ideal scientific mind, therefore, must always be held in a state of balance which the slightest new evidence may change in one direction or another. It is in a constant state of skepticism, knowing full well that nothing is certain." — HENRY A. ROWLAND.

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PREFACE

Since the last edition (1909) of Air, Water, and Food was published there have been distinct advances in analytical methods, and a changed point of view has brought about a somewhat different interpretation of results. This is particularly true with regard to the relation of air to health and comfort. At the present time the subject is still in a somewhat transitory state. In order that the book might remain useful it seemed necessary to make a careful revision of the whole.

The death of one of the authors, Mrs. Ellen H. Richards, made a change in authorship necessary. We are indebted to Prof. R. H. Richards for permission to use any material from the former edition. While realizing that the book was first written from a "missionary" standpoint (Mrs. Richards' strong point), it actually has been used mainly for college and technical school teaching; consequently the character of part of the general discussion has been considerably changed.

All of the discussion on air and water has been completely rewritten, as has the section on milk, the older methods revised, and numerous additions, to correspond with the latest practice, made. As in previous editions, these discussions are intended to be essentially elementary rather than exhaustive.

A. G. W.
J. F. N.

Boston, *July*, 1914.

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CONTENTS

Chapter		Page
I.	Three Essentials of Human Existence	1
II.	Air and Health	9
III.	Air: Analytical Methods	21
IV.	Water: Its Relation to Health, Its Source and Properties	43
V.	Safe Water and the Interpretation of Analyses	56
VI.	Water: Analytical Methods	69
VII.	Food in Relation to Human Life, Definition, Sources, Classes, Dietaries	111
VIII.	Adulteration and Sophistication of Food Materials	124
IX.	Analytical Methods	135
	Appendices	208
	Bibliography	228

AIR, WATER, AND FOOD

CHAPTER I

THREE ESSENTIALS OF HUMAN EXISTENCE

Air, water, and food are three essentials for healthful human life. Chemical Analysis deals with these three commodities in their relation to the needs of daily existence: first, as to their normal composition; second, as to natural variations from the normal; third, as to artificial variations — those produced directly by human agency with benevolent intention, or resulting from carelessness or cupidity. A large portion of the problems of public health come under these heads, and a discussion of them in the broadest sense includes a consideration of engineering questions and of municipal finances. This, however, is beyond the scope of the present work.

The following pages will deal chiefly with such portions of the Chemistry of Sanitation as come directly under individual control, or which require the education of individuals in order to make up the mass of public opinion which shall support the city or state in carrying out sanitary measures.

A notable interest in the subject of individual health as a means of securing the highest individual capacity both for work and for pleasure is being aroused as the application of the principles governing the evolutionary progress of other forms of living matter is seen to extend to mankind.

Will power may guide human forces in most economical ways, and may concentrate energy upon a focal point so as to seem to accomplish superhuman feats, but it cannot create force out of nothing. There is a law of conservation of human

energy. The human body, in order to carry on all its functions to the best advantage, especially those of the highest thought for the longest time, must be placed under the best conditions and must be supplied with *clean air*, *safe water*, and *good food*, and must be able to appropriate them to its use. The day is not far distant when a city will be held as responsible for the purity of the air in its schoolhouses, the cleanliness of the water in its reservoirs, and the reliability of the food sold in its markets as it now is for the condition of its streets and bridges. Nor will the years be many before educational institutions will be held as responsible for the condition of the bodies as of the minds of the pupils committed to their care; when a chair of Sanitary Science will be considered as important as a chair of Greek or Mathematics; when the competency of the food-purveyor will have as much weight with intelligent patrons as the scholarly reputation of any member of the Faculty. Within a still shorter time will catalogues call the attention of the interested public to the ventilation of college halls and dormitories, as well as to the exterior appearance and location.

These results can be brought about only when the students themselves appreciate the possibilities of increased mental production under conditions of decreased friction, such as can be found only when the requirements of health are perfectly fulfilled.

Of the three essentials, air may well be considered first, although its office is to convert food already taken into heat and energy. Its exclusion only for a few minutes causes death, and in quantity used it far exceeds the other two. Again, so important is the action of air that the quality of food is of far less consequence when abundant oxygen is present, as in pure air, than when it is present in lessened quantity, as in air vitiated by foreign substances.

Individual habit has much to do with the appreciation of good air, and as our knowledge of the value of an abundance of this substance in securing great efficiency in the human being increases, we shall be led to attach more importance to the sufficiency of the supply.

In northern climates air is not free to all in the sense of costing nothing, for the coming of fresh air into the house means an accompaniment of cold which must be counteracted by the consumption of fuel. A mistaken idea of economy leads householders, school boards, and college trustees to limit the size of the air-ducts as well as of the rooms. It is therefore necessary to emphasize the facts which science has fully established, in order to secure the survival of the fittest of the race under the present pressure of economic conditions, which take so little account of the highest welfare of the human machine.

Air, water, and soil are the common possessions of mankind. It is impossible for man to use either selfishly without injury to his neighbor and without squandering his inheritance. Primitive man could leave a given spot when the soil became offensive, and neighbors were then too few to require consideration; but neither man nor beast could with impunity foul the stream for his neighbor who had rights below him. The soil is permanent; one knows where to look for it and its pollution. Air is abundant and is kept in constant motion by forces of nature beyond human control, so that, save in the neighborhood of an exceptionally offensive factory, man does not often foul the free air of heaven; it is only when he confines it within unwonted bounds that it becomes a menace.

Water is the next precious commodity of the three. Without it man dies in a few days; without it the soil is barren; without it air in motion parches all vegetation and carries clouds of dust particles; without it there is no life. As population increases it becomes necessary to collect as much of the rainfall as possible, to store it until needed, and to use it with discretion. After use it is often loaded with impurities and sent to deal death and destruction to those who require it later, and yet, in nature's plan, it is the carrier of the world, and rightly treated and carefully husbanded there is enough for the needs of all. Its presence or absence has been the controlling force in determining the habitations of men. In its office of carrier it not only brings nourishment in solution to the tissues of the

human body, but also carries away the refuse material. It is a cardinal principle in all sanitary reforms to get rid of that which is useless as soon as possible. Too little water allows accumulation of waste material and a clogging of the bodily drainage system.

The average quantity needed daily by the human body is about three quarts. Of this a greater or less proportion is taken in food, so that at times only from a pint to a quart need be taken in the form of water as such.

Next in importance to quantity is the quality, dependent somewhat upon the uses to which it is to be put. As a rule, the moderately soft waters are the best for any purpose. For drinking purposes water must be free from dangers to health in the way of poisonous metals, decomposing matters, and disease-germs. For domestic use economy requires that it should not decompose too much soap. Manufacturing interests require that it should not give too much scale to boilers; for agriculture there should not be too much alkali.

From the nature of things, no one family or city can have sole control of a given body of water. Those on the highlands may have the first use of the water, which then percolates to a lower level and is used by the people on the slopes over and over before it reaches the sea to start again on its cycle of vapor, cloud and rain, brook and river. Although receiving impurities each time, there are many beneficent influences at work to overcome the evils resulting from this repeated use. That which is dissolved from one portion of earth may be deposited on another. As the plant is the scavenger of the air, withdrawing the carbon dioxide with which it would otherwise become loaded, so the water has also its plant life, purifying it and withdrawing that which would otherwise soon render it unfit for any use.

Pure water is found only in the chemical laboratory; the most that can be hoped for is that human beings may secure for themselves water which is safe to drink, which will not impair the efficiency of the human machine.

The importance of the third essential for human life, food, and the close interdependence of all three, may be clearly shown. Of little use is it to provide pure air and clean water if the substances eaten are not capable of combining with the oxygen of the air or of being dissolved in the water or the digestive juices; of less use still is it to partake of substances which act as irritants and poisons on the tissues which they should nourish, and thus prevent healthful metabolism and respiratory exchange.

And yet a large majority of those who have acquired some notion of the meaning and importance of pure air and are beginning to consider it worth while to strive for clean water pay not the least attention to the sanitary qualities of food; the palatable and æsthetic aspects only appeal to them.

Steam-power is produced by the combustion of coal or oil. Human force is derived by releasing the stored energy of the food in the body. The delicately balanced mechanism of the human body suffers even more from friction than the most sensitive machine, and the greatest loss of potential human energy occurs through ignorance, carelessness, and reckless disregard of nature's laws in regard to food.

It is necessary to know, first, what is the normal composition of a given food-material. This is found by analyses of many typical samples. Second, is the sample under consideration normal? To answer this requires an analysis of it, and a comparison of the results with standards. If it is not normal, in what way does it depart from the standard both in healthfulness and in quality? Third, if a food-substance is normal, what are its valuable ingredients and in what proportions are they to be used in the daily diet?

In regard to meat, milk, and fish, the sanitary aspect for the chemist resolves itself into two questions: Is the substance so changed as to become a possible source of poisonous products? Or has anything in the nature of a preservative been added to it? If so, is it of a nature injurious to man?

There is, however, a great range of *quality* in some of the most abundant foodstuffs, such as the cereals, especially in the

nitrogen content. This is most important to the vegetarian and to institutions where economy must be practiced. The following variations in the composition of leading cereals will illustrate:

	Water.	Nitrogenous substance.	Crude fat.	Carbohydrates.	Fibre.	Ash.
Oats, maximum.....	20.80	18.84	10.65	64.63	20.08	8.64
Oats, minimum.....	6.21	6.00	2.11	48.69	4.45	1.34
Oats, American hulled...	12.11	13.57	7.68	63.37	1.30	2.03
Corn, maximum.....	22.20	14.31	8.87	52.08	7.71	3.93
Corn, minimum.....	4.68	5.55	1.73	72.75	0.99	0.82

One sample of wheat flour may contain 14 per cent of nitrogenous substance, another may yield only 9. A day's ration, 500 grams, will give 70 grams of gluten, etc., in the one case and only 45 in the other. This difference of 25 grams would be a serious factor in the dietary of an institution where little additional protein is given, and it alone might be the cause of dangerous under-nutrition.

The next step would naturally be to determine how definitely these varying percentages mean varying nutrition. To this end a study of vegetable nitrogenous products in their combination or contact with cellulose, starch, and mineral matter is needed. Much work remains to be done before these questions can be even approximately answered.

At the low cost of one cent a pound, common vegetables yield only about one-fifth as much nutriment as one cent's worth of flour, yet they contain essential elements and deserve to be carefully studied.

The sanitary aspect of food demands a study of normal food and food value even more than of adulterants or of poisonous food, ptomaines and toxines. The cultivation of intelligent public opinion is most important, and each student should go out from a sanitary laboratory a missionary to his fellow men. That is, the office of a laboratory of sanitary chemistry should be so to diffuse knowledge as to make it impossible for educated

people to be deluded by the representations of unprincipled dealers. Freedom from superstition is just as important in this as in the domain of astronomy or physics. So long as chemists are employed by manufacturing concerns in making adulterated and fraudulent foodstuffs, so long must other chemists be employed in protecting the people until the public in general becomes wiser. A part of the common knowledge of the race should be the essentials of healthful living, in order that the full measure of human progress may be enjoyed.

There is needed a greater respect for food and its functions in the human body, a better knowledge of its effect on the daily output of energy, its absolute relations to health and life, and the enjoyment of the same. The familiarity with these facts which is given by a few hours' work in the laboratory will make a lasting impression and will enable the student to benefit his whole life, even if he never uses it professionally. It is purely scientific knowledge, just as much as that derived from a study of the phases of the moon or the formulæ of integration.

The variety of operations in such work, calling for great diversity of apparatus and methods, is an educational factor not to be overlooked in laboratory training.

For all detailed discussions and methods the reader is referred to such works as those of Wiley, Allen, Leach, etc., but for the student who needs to study, as a part of general *education*, only typical substances, and such methods as can be carried out within the limits of laboratory exercises in a college curriculum, the following pages are written. Not enough is given to frighten or discourage the student, but enough, it is hoped, to arouse an interest which will impel him at every subsequent opportunity to seek for more and wider knowledge.

Food is too generally regarded as a private, individual matter rather than as a branch of social economy; it is, however, too fundamental to the welfare of the race to be neglected. Society, in order to protect itself, must take cognizance of the questions relative to food and nutrition.

Formerly each race adapted itself to its environment and

trained its digestion in accordance with the available food supply. In America to-day the question is not how to get food *enough,* but how to choose from the bewildering variety offered that which shall best promote the health and develop the powers of the human being, and, what is of equal importance, how to avoid over-indulgence, which weakens the moral fibre and lessens mental and physical efficiency. In spite of all preaching, few really believe that plain living goes with high thinking. Professor Patten says that the ideal of health is to obtain *complete* nutrition. Over-nutrition as well as under-nutrition weakens the body and subjects it to evils that make it incapable of survival.

No other form of social service will give so full a return for effort expended as the help given toward better diet for children and students. Fortunately help is coming fast. The United States Government is giving much study to food problems, and by publications is making available the work of other countries. The later bulletins listed in the bibliography at the end of this volume are especially valuable. What is now needed is a general recognition of the importance of the subject.

CHAPTER II

AIR AND HEALTH

The air we breathe is a mixture of various gaseous substances containing more or less finely divided solid particles. What may be called "pure" air contains 20.938 per cent * by volume of oxygen, 0.031 per cent of carbon dioxide, 78.09 per cent of nitrogen, 0.94 per cent of argon and other rare gases belonging to the argon group.

All the air with which we actually have to deal contains also varying amounts of moisture, expressed in terms of "relative humidity." Air at a low temperature can hold much less moisture than at a high temperature. For example, one cubic foot of air at 20° F. will hold 1.235 grains of water vapor, while at 70° 7.98 grains will be held. The relative humidity is the ratio of the amount of moisture which the air actually contains to the amount which it could hold at the same temperature if completely saturated. As water vapor is lighter than dry air, the higher the humidity the less will a given volume of air weigh. This effect is familiar in the action of a barometer which falls on the approach of a rain storm, — the reading on such an instrument being dependent on the weight of air above it.

Besides moisture, the air in cities may contain a variety of substances such as ammonia, sulphur dioxide, sulphur trioxide, etc., and almost always dust, bacteria, yeasts, and molds. Samples of air † taken in the down town districts of New York and Boston showed at the street level numbers of dust particles per cubic foot of air varying from 170,000 to 500,000, the num-

* Benedict, Composition of the Atmosphere. Carnegie Institution, Publication No. 166.

† G. C. Whipple and M. C. Whipple, *Am. J. Pub. Health*, 1913, **3**, p. 1140.

ber gradually decreasing as the height above the street increased, until only about 27,000 were found in the air taken from the fifty-seventh floor of the Woolworth Building, 716 feet high. In a house, school room or public building the numbers of dust particles are equally variable, with a tendency to be somewhat higher, depending on the location of the building, and whether or not the air entering is purified. Thus in an investigation of the air of school rooms,* few cases were found where the numbers were less than 200,000 per cubic foot, and they varied from this to over 1,500,000, the greater proportion being between 200,000 and 600,000, much higher than is generally found in outdoor air. The numbers of bacteria found in the air are small compared to the dust particles, there being about $\frac{1}{200}$ as many in outdoor air, and even less in indoor air, in 85 per cent of the samples taken in school rooms † the number of micro-organisms being less than 150 per cubic foot. In country districts the numbers of both dust particles and bacteria in the air are extremely small.

Under ordinary conditions the presence of dust and bacteria has no particular significance. In fact it is the opinion of most sanitarians that the danger of the spread of disease by the carrying of bacteria through the air is small, the contact necessary for this to happen being much closer than generally exists in offices and schoolrooms. There are certain special cases where dust particles may be harmful, — such as the dust consisting of small particles of metal found in certain factories, and the organic dust found in the air in certain rooms in textile mills. Some of these dusts, such as white lead, are themselves actually poisonous to the system, while others lodge in the lungs and lower the vitality so that pneumonia and tuberculosis are more liable to gain a footing.

Poisonous gases are occasionally found in air, — the most important being carbon monoxide which comes from leaky gas jets or pipes, or from a defective furnace. As this gas has al-

* Winslow, *Am. J. Pub. Health*, 1913, **3**, p. 1158.
† Winslow, *loc. cit.*

most no odor, insensibility may occur without the victim realizing what is taking place. For this reason it has been found necessary, where this gas is used for lighting, to require the introduction into it of some substances with strong odors. Carbon monoxide acts as a poison by combining with the hæmoglobin of the blood, and preventing the absorption of oxygen.

In the air of mines, methane, — or fire damp as it is called, — is sometimes present. This forms an explosive mixture with oxygen, and is frequently the cause of mine explosions.

Respiration. — External respiration consists of alternately filling and emptying the lungs. In the lungs, oxygen, breathed in with the air, is exchanged for carbon dioxide brought to the lungs by the blood. The blood leaving the lungs contains oxygen which is carried to all parts of the body, and passes * from the blood in the capillaries into the tissues where oxidation takes place. The carbon dioxide formed passes back into the blood and hence into the lungs. Expired air, therefore, contains less oxygen and more carbon dioxide than inspired air. An average composition would be, — oxygen, 16.03 per cent; carbon dioxide, 4.38 per cent; nitrogen, etc., 79 per cent.

The process of exchange of oxygen and carbon dioxide in the lungs is partly a physical one, — that is, the vapor pressure of oxygen is greater in the lungs than in the blood, and, therefore, oxygen passes from the former to the latter. With carbon dioxide the reverse is true. Therefore, if air high in carbon dioxide is breathed into the lungs this will increase the vapor-pressure of this substance, and hinder the elimination of it from the blood. But it appears to be impossible to account for the interchange of gases on a purely physical basis, and, therefore, it is thought that enzymes, which aid in the interchange, are at work.

Comfort. — The first two theories that were advanced to account for effects of discomfort when a room becomes "close" were based on the supposition that the products of respiration were poisonous when taken back into the lungs. In one theory

* See Hammarsten-Mandel. "A Text-book of Physiological Chemistry."

this poisonous substance was supposed to be carbon dioxide. That animals cannot live in an atmosphere composed of nitrogen and carbon dioxide, and that oxygen is necessary has long been known, but it was thought that carbon dioxide had a specific poisonous action and, therefore, should be present in any air used for human beings, in only very small amounts. This theory has been entirely disproved and carbon dioxide can no longer be regarded as in itself poisonous. If too much of the oxygen in the air becomes displaced by carbon dioxide it is impossible for animals to utilize the oxygen left, but this only happens when the oxygen content decreases to about 12 per cent. Practically such a low per cent is never found, as interchange of the air between a room and the outside is continually going on around windows and through walls. If, however, the oxygen is allowed to remain at about 21 per cent, very large quantities of carbon dioxide may be present without any ill effects. Experiments have shown conclusively * that carbon dioxide cannot be blamed for discomfort in a crowded hall or theatre.

The other theory, — known as the "crowd poison" theory was based on some experiments which seemed to show that organic poisons were given off during respiration, and that these substances were the cause of the headaches and nausea sometimes experienced by sensitive persons in "close" rooms. At the present time there are some adherents to this theory, but there has been little real evidence produced in its support. The first proofs of the non-poisonous character of exhalations were obtained by Formanek in a long series of experiments † and more recently Winslow ‡ using the principles of anaphylaxis failed to obtain any results which showed the presence of the poisons (or toxins) in expired air.

At the present time it is quite generally believed that sen-

* See Crowder. "Ventilation of Sleeping Cars." *Arch. Intern. Med.*, 1911, **7**, pp. 85–133.

† Archiv für Hygiene, 1900, **38**, p. 1.

‡ *Loc. cit.*

sations of comfort and discomfort are dependent upon the rate of loss of heat from the body. If this is normal, then comfort results, if either too high or too low, then discomfort, headaches and nausea may follow. Just what this heat loss should be, measured in any system of units, is not known, but certain of the methods by which the loss takes place, and the factors which influence the rate may be discussed.

There are three ways by which heat can be transferred from the body to the surrounding atmosphere. (1) Evaporation. — The change from the liquid to the gaseous state is accompanied by an absorption of heat. Thus when water evaporates from the surface of the body, heat is removed with it. (2) Transmission (by conduction and convection). Heat passes from a warm to a cold body when the two are in contact. For the greater part of the year the animal body is warmer than the atmosphere, and, therefore, the latter is continually receiving heat from the body. Since warm air rises, convection currents may be set up carrying away the heat already given up to the air. (3) Radiation. — The first two methods depend directly on the presence of matter. In radiation heat is transferred in all directions by means of ether waves, and the medium through which the radiation takes place does not necessarily become heated. There is no data available on the loss of heat from the body in this way, and we do not know what part it actually plays in comfort.

These three methods by which heat may be given off from the body may be acting simultaneously, — in fact they generally are doing so, — and one or more may be negative in its action, — that is may be supplying heat to the body. Further, while they act entirely independently of each other, they are each influenced by the same conditions of the atmosphere, and it is these physical conditions which are the ones capable of regulation, and which determine good or bad ventilation. These are, — temperature, humidity and motion.

Temperature. — Temperature affects evaporation, because the higher the temperature of the air the more moisture is it capable

of taking up. It affects conduction, because the greater the difference of temperature between two bodies the greater the amount of heat passing from that at the higher to that at the lower temperature. It affects convection, because convection currents are started by warm air rising and cooler air taking its place.

Humidity. — Heat loss by evaporation is more dependent on humidity than on any other factor. Relative humidity is a measure of the per cent saturation of the air by water vapor, and it is obvious that the higher the humidity the less will be the opportunity for the air to take up more moisture, and, therefore, the less rapid the evaporation from the body. Transmission of heat from the body is affected by the humidity, because moist air is a better conductor than dry air, and, therefore, the higher the humidity the greater the rate of heat conduction. (Relative humidity, as can be seen from a foregoing discussion, is itself affected by the temperature.)

Motion. — The motion of the air influences evaporation by carrying away from the body more or less rapidly the air which has become completely saturated with moisture, and thus allowing access to unsaturated air. If the air and the body are perfectly quiet evaporation will be gradually retarded until it is nearly zero. Convection currents are movements in the air started by differences in temperature. These movements will be greatly increased by any motion in the air, and, therefore, the greater the motion the more rapid will be the transference of heat in this way.

It is important to remember that these three factors, temperature, humidity and motion, — are always acting simultaneously, and that there may be an increase in the rate of heat loss above the normal by one or more of them at the same time that the rest tend to decrease this rate. Furthermore, the same factor, humidity for example, may tend to increase the heat loss above the normal by one method, — perhaps by evaporation, — while at the same time, the same degree of humidity may tend to decrease below the normal the heat loss by another method,

perhaps by transmission. The degree of comfort felt under any specified conditions is, therefore, the resultant of all effects, some tending to increase and others to decrease the rate of heat loss from the normal.

This can be readily illustrated. Suppose that the temperature is 95° F., the humidity 90 per cent and there is but very little motion in the air. The result is well known, — a feeling of heaviness and considerable discomfort. Why?

(1) The high temperature allows the air to take up a considerable amount of moisture, thus tending to increase the heat loss by evaporation, with the consequent cooling effect on the body. On the other hand, the heat loss by conduction, convection and radiation are only very small as they depend on the difference of temperature of the body and the air.

(2) The high humidity prevents the rapid evaporation of moisture, and, therefore, tends to decrease the heat loss from the body. This more than counteracts the increased capacity of the air for moisture, due to the high temperature. On the other hand, the high humidity makes the air a better conductor of heat, and, therefore, tends to increase the heat loss by conduction. This, again, is counteracted by the high temperature, temperature being the more important factor in this method of loss.

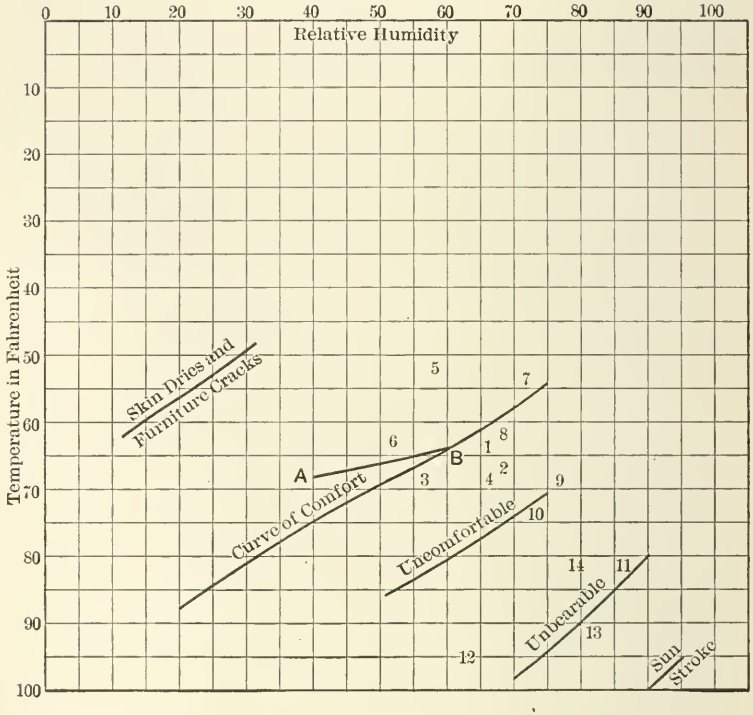
(3) The very slight motion of the air tends to decrease the heat loss by evaporation and convection.

The net result is that heat does not leave the body as rapidly as it should, and we feel hot and uncomfortable.

Application of this theory of regulation of loss of heat is not wholly adequate to explain all conditions. Another factor seems to be involved, that of loss of moisture, apart from any loss of heat which accompanies this. "Probably much of the harm attributed to damp and to cold is due to diminished water circulation, etc."* With this added factor it is possible to explain most of the uncomfortable conditions. The uncertainty of the theory lies in the fact that we have been unable to test it

* Macfie, Air and Health.

THE CURVE OF COMFORT



Mean annual temperature and humidity of health resorts:

1	Algiers	5	Arequipa
2	Alexandria	6	Luxor-winter
3	Cairo	7	Los Angeles
4	Bermuda	8	Madeira

Unfavorable to white man's residence:

9	New Orleans	12	Persia
10	Havana	13	India
11	Malay Archipelago	14	Singapore

A-B Most comfortable for indoor workers (Hill).

experimentally and to determine the exact heat loss due to each factor.

Hill * has plotted a series of curves which are intended to represent the various conditions of comfort in terms of temperature and humidity. Thus it is seen that a temperature of 55° F. and a humidity of 70 per cent gives comfort, and as the temperature increases the humidity must be decreased. At 68° F., the temperature generally desired in the house, the humidity must be around 50 per cent.

Ventilation. — In ventilating a public building or a house, it is necessary to supply a sufficient quantity of air in the proper condition. In most cases this condition is, that the air in the room shall be at a temperature of 68° to 70° F., and with a humidity of 50 to 70 per cent. As long as the humidity does not go too high, it seems to be a secondary factor so far as health is concerned. More discomfort is felt from overheating than from any other cause. This is also true in many factories, but there are some where high humidity must be considered, such as is necessary to maintain in connection with certain textile operations. It should be remembered that the higher the temperature the more sensitive does one become to high humidity.

Another condition which must be met in ventilation practice is that governed by the carbon dioxide content of the air. As pointed out above, this substance is not itself poisonous, but it is useful in serving as an index of the amount of unused air being supplied. The normal individual gives off from 0.6 to 0.8 cubic feet of carbon dioxide per hour, and this will gradually accumulate in a room unless the air is continually being replaced. The amount of carbon dioxide present in a room can, therefore, be used to determine whether or not there is sufficient replacement of used air by fresh air. The allowable amount of carbon dioxide is about 10 parts per 10,000 of air. Amounts above this may be allowed in certain special cases where the carbon dioxide does not come from man or animals. If only 6 or 7 parts are present, the ventilation may be considered excellent. In order

* Hill, Recent Advances in Physiology and Biochemistry.

to accomplish this about 2000 cubic feet of fresh air per person per hour must be supplied. The amounts actually recommended depend somewhat on the use to which the room or building is to be put, these amounts varying between 1000 cu. ft. for a waiting room and 2500 for a hospital. Where it is difficult to determine how many people will be present the calculations may be based on the number of complete changes of air per hour, these being from one to five in a residence, and from one to two in an auditorium.*

It is also possible to calculate from analytical data the interchange of air going on under given conditions, and thus test the efficiency of a ventilating system. If, after a room has been occupied and the occupants removed, the air is analyzed for carbon dioxide, the room allowed to remain a definite length of time, and another analysis made, the interchange may be calculated from a formula given by Barker: †

$$V = \frac{C}{T} \log \left(\frac{k_1 - \alpha}{k_2 - \alpha} \right)$$

where C is the contents of the room in cubic feet, T the time in hours between the original amount of carbon dioxide k_1 in one cubic foot of air, and the final amount k_2 in one cubic foot of air, α the proportion of carbon dioxide in one cubic foot of pure atmospheric air, and V the interchange in cubic feet per hour.

Ventilation depends on the movement of air currents in such a way as to continually supply fresh air and to remove used air. This must be done so that no drafts will be felt at any part of the room. The system actually used will depend on the kind of building and room, — as well as on the kind of heating used. In the ordinary dwelling house ventilation is almost always left to look after itself. Even in the best built houses there is going on constantly an interchange of air around the windows and doors. This is not sufficient on winter evenings

* Greene, "Elements of Heating and Ventilation," p. 23.

† Baker, "The Theory and Practice of Heating and Ventilation," p. 164. A number of other useful ventilating formulæ are also given.

when kerosene or gas lamps are burning, and most rooms soon become stuffy. To aid this natural ventilation, windows, open fire places and hot air furnaces are used. Excellent results may be obtained from the careful use of the open window, but it requires considerable time as well as care to operate them so that no drafts will result. Where a hot air system of heating is used a house may be well ventilated, — the air which is forced in through registers going out after proper circulation, through ventilators or around windows. Care should be taken to place registers to get this circulation.

In a large building, — office, educational, or auditorium, — the problem is somewhat different. Here it is useless to depend on natural ventilation and some artificial means must be employed. There are two general methods of air circulation in use, upward and downward. Both have their advantages and disadvantages. Upward ventilation would seem theoretically the best, as expired air, being warm, rises and creates an upward current, which can be easily drawn into an outlet. This system can be used, but it presents certain difficulties. The first is that unless air comes into the room through a very large number of small holes in the floor, drafts of cold air around the feet are certain to be felt. This would only be practical in an auditorium with stationary seats. Besides, objection is sometimes made that odors from the clothing are made more noticeable by being carried past the nose. The reverse system, downward ventilation, seems to be more practical. Here the air is introduced from the ceiling and is drawn out through ducts in or near the floor. More often air is introduced from the walls of the room. In this case it is necessary to so arrange the inlet and outlet that air from the former will circulate around the room before reaching the latter. To do this, the outlet is generally placed a little below the inlet on the same wall, this being on the cold side of the room. The air may be forced into the room under pressure from a fan, called the plenum system, or may be drawn out from the room by a fan in the outlet, called the vacuum system.

In cities where there is necessarily much smoke and dirt, it may be considered best to purify, in some way, the air entering a building. The simplest method is to screen the incoming air through fine wire gauze or cheese cloth. A more effective way is by means of air washers. All of these, and there are a number of them on the market, depend on the passage of the air through a spray of water which removes dirt, bacteria and soluble substances. Since these machines spray water into the air they are also humidifiers, and may be used as such, particularly in textile factories where it is necessary to carry on certain operations in moist air.

It is also possible to take air out of a room, wash it, cool it and send it back into the same room.* This would effect a saving of coal if it were practical to operate.

Another method which has been in some use for purifying air is by means of ozone. During the last year there has been much discussion on this subject,† and very serious doubts have been thrown on the real usefulness of this method. That ozone in the presence of a large amount of moisture is a good disinfectant cannot be denied, but under the dry conditions of the atmosphere its germicidal effect is small. However, in most cases it is not bacteria which we need to kill, but odors. On this point the evidence is not quite so clear. Most are agreed that the odors disappear, but it is still a question whether the substances producing them are actually destroyed, or whether the odors are masked by those of ozone. From a standpoint of health, this would also be immaterial if it could be proved that the ozone itself was harmless to breathe. At the present time the evidence seems to be the other way.

* See article on Recirculated Air. McCurdy, *Am. Phys. Ed. Rev.*, Dec., 1913.

† Jordan and Carlson, *J. Am. Med. Assn.*, 1913, **61**, pp. 1007–1012; Norton, *Eng. Rec.*, 1913, **68**, p. 732; Vosmaer, *J. Ind. Eng. Chem.*, 1914, **6**, p. 229.

CHAPTER III

AIR: ANALYTICAL METHODS

In an investigation of the air of any room or public building it is not enough to make one or two observations, as these might be entirely misleading, but a sufficient number must be taken to get a fair estimate of the conditions. Thus in a room, readings of the physical instruments must be made and samples for chemical analysis taken at a number of points, and these repeated at intervals of five or 10 minutes until six or eight have been taken. Slight changes constantly occur which are not of any importance in practical work, but fortunately most of the instruments and most of the methods used are not delicate enough to be influenced by changes of this character. In short, it is average conditions which are of importance, and which should be recorded.

Physical Determinations. — *Temperature.* — The use of the thermometer is too well known to need any detailed statement. Mercurial thermometers are the most accurate for practical work, but care should be taken that the bulb of the thermometer is suspended in the air and not placed against a wooden back, as in the latter case the reading lags behind the actual changes in the temperature of the air. Where it is desired to have a continuous record, recording thermometers are to be recommended. These depend on the contraction and expansion of a metal combination, with the changes of temperature, the metal being connected with a pen which records the changes on a paper disc moved by clockwork.

Pressure. — Air pressure is measured by barometers, of which there are two types, — mercurial and anaeroid, both of which are well known. Since the barometric reading depends on the weight of the column of air above the instrument, the reading

will vary with the distance above sea level, and with the composition of the air. In the latter case the only important factor is moisture. As water vapor is lighter than dry air, the larger the moisture content the lighter the moist air and the less the pressure. Thus a low barometric reading indicates the approach of a storm.

Humidity. — Relative humidity has already been described. The most accurate method of measurement is by means of wet and dry bulb thermometers. The rate of evaporation of water into air at any one temperature depends on the amount of moisture already present. Since evaporation is accompanied by absorption of heat, the surface from which the water evaporates will be cooled in proportion to the rate of evaporation. If the bulb of a thermometer is surrounded by a film of moisture, which can readily be done by means of a piece of cloth or wick with one end dipped in a reservoir of water, this cooling can be measured by the lowering of the temperature below that of a thermometer whose bulb is surrounded by air alone, and the lowering is proportional to the relative humidity. In the appendix will be found a table from which the relative humidity can be obtained from the reading of the dry and wet bulb thermometers. In order that the wet bulb thermometer may come quickly to equilibrium an instrument called the psychrometer has been devised for rapidly rotating the thermometers.

Another method of measuring the humidity is by means of the hair hygrometer. In this instrument a number of horse hairs are placed under tension by means of a small weight. The distance to which the hairs will be stretched will depend on the amount of moisture taken up from the air, — the higher the moisture the greater the stretching. The weight can be readily connected to an indicator which will record the relative humidity on a dial, or a pen can be attached, to make a recording instrument, in a similar manner to that used with a recording thermometer.

Motion. — Where the velocity of air is considerable, as in the case of wind or in such places as ventilation ducts, measure-

ments can be made by the use of anemometers. However, in a room, the movement of air is much too slow, and the direction of currents too varied, for such an instrument to be of use. The best method is by use of smoke from a joss stick or cigar.*

Dust. — The simplest method for determining dust in air is to draw a measured quantity of air through a weighed tube containing a cotton plug. For this it is necessary to have a suction pump, — the variety which may be attached to a water faucet is useful, — a meter, such as a gas meter, and a tube containing a cotton plug. The tube with the plug should be dried in a desiccator before each weighing as moisture may be absorbed from the air passed through. Knowing the amount of air and the increase of weight of the cotton filter, the amount of dust per unit volume of air can be calculated. Where the amount of dust is large, the cotton plug can be replaced by one of granulated sugar. The amount of dust is then determined by dissolving the sugar in water and then filtering through a weighed Gooch crucible.

The most accurate determinations of dust particles can be made by means of the "Dust Counter" or the "Koniscope." Both of these instruments † are too expensive to be very generally used.

An apparatus for taking dust samples of air has recently been described by Baskerville,‡ and would seem to be useful and sufficiently accurate for practical purposes.

Chemical Determinations. — The first systematic study of the atmosphere was made by Scheele, in 1779, shortly after the discovery of oxygen. Since that time more and more accurate methods have gradually been developed, culminating in that used recently by Benedict.§

* Shaw, "Air Currents and the Laws of Ventilation." Cambridge, 1907.

† See "Standard Methods for the Bacterial Examination of Air," *Am. Pub. Health Assn.*, 1910, p. 38.

‡ *J. Ind. Eng. Chem.*, 1914, **6**, p. 238.

§ For a detailed history of air analysis see Benedict, "The Composition of the Atmosphere with Special Reference to its Oxygen Content," Carnegie Institution of Washington, 1912, Publication No. 166.

In practice the only chemical test made on air is that for carbon dioxide. In cases of poisoning, tests may be made for carbon monoxide or methane, and in experiments with respiration, oxygen determinations together with those for carbon dioxide are considered necessary.

The methods for the determination of carbon dioxide are all based on absorption by alkalies, the amount of this absorption being measured either by direct determination of the diminution of a given volume of air, or by determination of the amount of alkali used for the absorption.

Collection of Samples. — Methods for collecting samples of air for chemical analysis will vary somewhat with the method and apparatus used. In certain cases the sample is measured directly into the analytical apparatus, while in others, — and these are the more practical methods, — the sample is first collected in a balloon or bottle. Where large amounts are needed, as in the Pettenkofer method, the samples are collected in a four- or six-liter bottle, the volume of which has been determined by weighing both empty and filled with water. The bottle is fitted with a two-hole rubber stopper with a short piece of glass tubing to serve as an inlet in one hole and a long brass tube extending to the bottom of the bottle, in the other hole. This brass tube is connected to a bellows with the valves arranged so that air will be drawn out of the bottle. Pumping should be continued until the air originally in the bottle has been entirely replaced, which will take from 30 to 50 strokes of the bellows. The stopper and tube are then removed, and the bottle closed with a stopper as described on page 34.

For the Walker and the Cohen and Appleyard methods a much smaller volume is all that is needed, — from 500 c.c. to two liters. The simplest method is to fill the bottle with water and pour it out. This has the disadvantage that expired air from the collector may reach the bottle.

A better method is to fit two bottles each with a 2-hole rubber stopper. In one hole of the stopper of bottle (*A*) (Fig. 1) insert a short piece of glass tubing, and in the other a longer

piece of tubing extending nearly to the bottom of the bottle. In the stopper of (*B*) insert a short piece of glass tubing just reaching through the stopper, and a longer tube extending nearly to the bottom, and fitted with a piece of small bore rubber tubing and a pinch clamp. Connect the short tube of bottle (*B*) with the long tube of (*A*) by means of a rubber tube and close with a pinch clamp. Fill (*B*) with recently boiled water, open clamp (*a*), close clamp (*b*) and insert stopper with connections, into the bottle. Then close (*a*). Invert (*B*) at the point at which the sample is to be taken. Release the pinch clamp (*a*), and then open the clamp (*b*). The bottle filled with air (*B*) is then closed with a solid rubber stopper and is ready for analysis. If bottle (*A*) is larger than (*B*) it can be used, together with the water, for taking a number of samples of air.

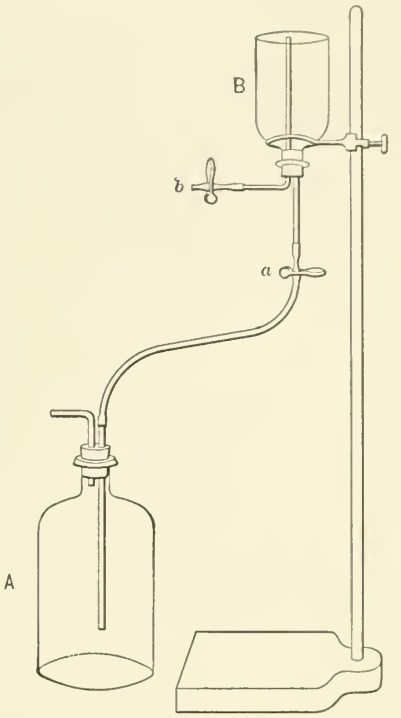


FIG. 1.

Another method by which sampling is made easier, but which does not give such accurate results, is the steam vacuum method. The apparatus is set up as in Fig. 2. Steam is supplied from a two-quart oil can nearly filled with water, or if preferred, from a liter flask. A rubber tube and piece of glass tubing connects the steam can with the inverted bottle, the size of which depends on the method of analysis used, the tube extending to within an inch of the bottom of the bottle. The bottles are made for ground glass stoppers, but are fitted with rubber

stoppers to which have been applied a thin coating of vaseline. Too much vaseline should be avoided, as it prevents the stopper staying in after the sample has been collected. The rubber stoppers should be one size larger than would ordinarily be used.

To prepare the bottle, fill the can two-thirds full with water, and boil for a few minutes to expel carbon dioxide and air. In-

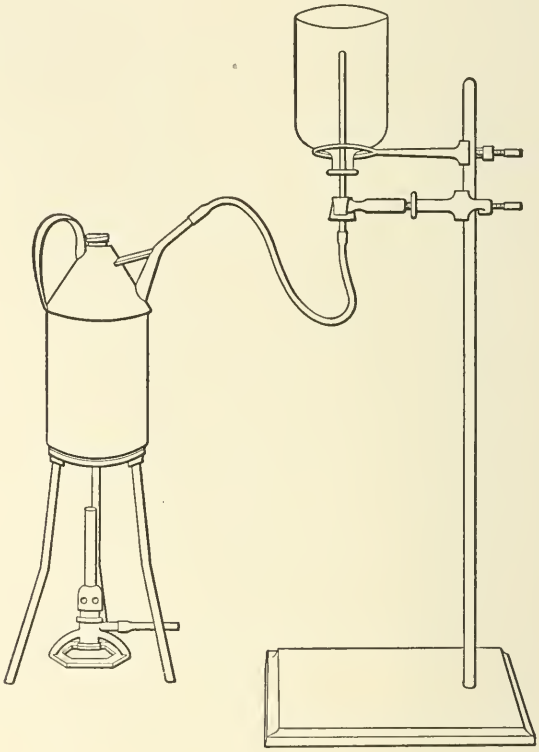


Fig. 2.

vert the empty bottle over the end of the tube, and allow to remain for three minutes. Keeping the bottle inverted, remove it from the tube, and quickly insert the rubber stopper. The stopper may be pushed in more securely by holding it against the table with a slight pressure, and keeping it there until the vacuum starts to form. When cool, the stopper should

project at least one-half an inch in order to be easily removed. A number of bottles can be prepared in the laboratory, and quite easily transported. All rubber stoppers which are used should first be boiled in dilute caustic soda, then in a dilute solution of potassium bichromate and sulphuric acid and thoroughly rinsed.

To collect the sample it is necessary only to remove the stopper, taking care to hold the bottle away from the face in order to prevent contamination from the carbon dioxide of the breath.

At the time of collecting the samples the following observations should be recorded: room, date, time, weather, place in room, number of people present, number of gas jets or lamps burning, number of doors, windows and transoms, methods of heating and ventilation, and anything else which would tend to influence the amount of carbon dioxide present.

In collecting samples, care must be taken to avoid currents of air or the close proximity of people. Exact duplicate analyses can be obtained only in empty or in nearly empty rooms. Even two sides of the same room will probably show differences, but two samples taken carefully side by side ought to agree within 0.05 part per 10,000.

Carbon Dioxide. — The most accurate analyses of air have been those obtained by Benedict by means of an apparatus especially designed by Dr. Klas Sonden.* The analysis depends upon the measurement of the decrease in volume of a sample of air after contact with a caustic alkali solution. Another accurate apparatus on the same principle is that of Pettersen-Palmquist, † which has been modified by Rogers, ‡ and more recently by Anderson.§ In all of these forms the manipulation is rather delicate, the apparatus is bulky to transport, and when obtained, the results are much more accurate than is necessary for any practical work.

* A description of this will be found in Publication No. 166, Carnegie Institution of Washington, already referred to.

† For description see Rosenau, "Hygiene and Preventive Medicine."

‡ See catalogue of Eimer and Amend.

§ *J. Am. Chem. Soc.*, 1913, **35**, p. 162.

Walker Method. — The method to be most recommended for practical analyses for carbon dioxide is that proposed by Walker.* It has been carefully studied in this laboratory † and slightly modified. The results are accurate to tenths of a part per 10,000.

Principle. — To a definite volume of air, usually one to two liters, is added a measured amount of standard barium hydroxide, care being taken to avoid contact of the solution with the air. After the absorption of the carbon dioxide, the solution is filtered under reduced pressure through asbestos and the clear barium hydroxide received into a known excess of standard hydrochloric acid. The absorption bottle is rinsed out with water free from carbon dioxide. The excess of acid is then determined by titration with barium hydroxide. It is essential for the complete absorption of the carbon dioxide that the barium hydroxide be largely in excess, so that not more than one-fifth of it is neutralized; furthermore, the absorbing solution must be shaken with the air for a considerable time.

Reagents and Apparatus. — The standard solutions used are N/50 hydrochloric acid, and barium hydroxide, approximately N/100, its exact strength relative to the acid being found daily by titration. It will be found advantageous to use solutions of this strength, somewhat more dilute than those recommended by Walker, on account of the increased accuracy with air nearly free from carbon dioxide. The decreased range of usefulness is readily compensated by the employment of smaller samples of the impure air.

The barium hydroxide is preserved with especial care. The hard-glass bottle containing it, placed on a high shelf so that the measuring apparatus can be filled directly by gravity, is heavily coated on the inside with barium carbonate. The bottle is closed by a rubber stopper with two holes, one of which carries the siphon tube dipping to the bottom of the bottle and supplying the measuring burette, while the other carries a fairly large glass *T*. (Fig. 3).

* *J. Chem. Soc.*, 1900, **77**, p. 1110.

† Woodman, *J. Am. Chem. Soc.*, 1903, **25**, p. 150.

From one-half the horizontal arm of this projects a glass tube carrying the device for protecting the solution. This device is shown drawn on a somewhat larger scale in the same sketch. The horizontal tube enters the T tube far enough to support the apparatus. Connection is made by a closely-fitting rubber tube. The longer tube, reaching nearly to the bottom of the test-tube, carries a fairly good-sized calcium-chloride tube which contains soda-lime, enclosed in the usual manner by plugs of cotton. The test-tube contains five to 10 c.c. of dilute (about N/50) caustic potash colored with phenolphthalein, the whole serving to indicate the efficiency of the soda-lime. From the other end of the horizontal arm of the T projects, in the same way, a long tube bent at right angles fitting by a rubber stopper into the top of the burette, thus making the whole a closed system, much after the manner of Blochmann.* Any air entering the bottle when the solution is drawn from the burette or when the burette is filled again must have come through the protecting apparatus. This will be found efficient if care is taken in the selection or preparation of the soda-lime.†

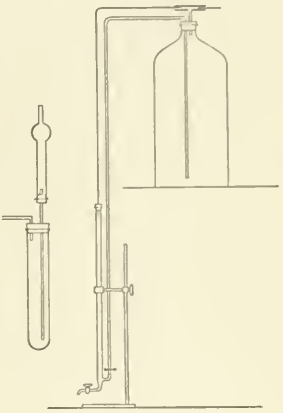


FIG. 3.

The burette used for the barium hydroxide is a glass-stoppered one, differing somewhat from the ordinary form. The portion below the graduations is narrowed and bent at a right angle. This horizontal part is fitted with an ordinary glass stop cock. This gives no trouble when kept well vaselined. The tip of the burette is kept covered with a little rubber cap when not in use, to prevent clogging from the formation of carbonate. The apparatus could easily be arranged with a

* *Ann. Chem.*, (Liebig), 1887, **237**, p. 39.

† Directions for preparing a good quality of soda-lime are given by Benedict and Tower, *J. Am. Chem. Soc.*, 1899, **21**, p. 396.

special pipette for the delivery of a definite charge of a baryta solution.

The apparatus used for filtering off the barium carbonate is shown in Fig. 4. On the base of a ring stand is placed an ordinary filter bottle of about 250 c.c. capacity closed by a rubber stopper with one hole. The suction pump is connected with the tube on the side of the bottle. A Gooch filtering-funnel, the upper part of which is cut off so that the remainder above the constriction is about an inch long, is put through the rubber stopper. The tip projecting into the bottle is bent so that the liquid shall flow down the side and not spatter. A rather close coil of stout platinum wire placed above the narrow portion serves as a support for an asbestos filter. A two-cm. Gooch filter plate serves as well as the platinum wire. In the upper part of the tube is a tightly-fitting rubber stopper, through which passes a narrow glass tube extending to within one-eighth inch of the asbestos layer, and provided above the stopper with a stop cock. Connection is made with the short tube of the inverted bottle by means of a rubber tube about 4 inches in length.

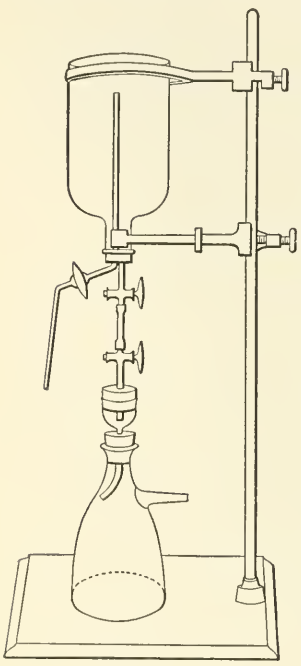


Fig. 4.

The inverted bottle is a carefully calibrated one of about one liter capacity, and is used for collecting the sample, the method preferably being by water displacement as described on page 25. Record the temperature and barometric pressure at the time the sample is taken. After collecting the sample the bottle is closed by a solid rubber stopper. For filtering, this is replaced by a rubber stopper through which pass two glass tubes. The longer tube reaches nearly to the bottom of the

bottle, is bent as shown, and contains a glass stop cock. The shorter tube ends internally just flush with the stopper, and outside is fitted with a stop cock and projects just far enough to make connection with the rubber tubing. The glass stop cocks may be replaced by rubber tubing and Mohr pinch clamps.

The filter is made of washed asbestos, free from acids, in the manner usual for Gooch crucibles. The same filter will do for a number of determinations. The asbestos layer should be about one-eighth of an inch thick and should be washed with distilled water.

Procedure. — Remove the stopper from the calibrated bottle containing the sample of air, and run in rapidly from the burette about 25 c.c. of the barium hydroxide solution, the exact amount being determined from the burette readings. Immediately replace the rubber stopper, place the bottle on its side and shake at very frequent intervals for 20 minutes, giving a sort of rotating motion so that the solution will spread over the bottle, and thus expose a large surface for absorption of the carbon dioxide.

While the absorption is going on prepare the filter (although it is better to prepare this, and to standardize the barium hydroxide before starting the determination) and also make about 100 c.c. of wash water for each determination. This latter is done by adding to distilled water one c.c. of a 10 per cent barium chloride solution and three drops of phenolphthalein, then titrating with the barium hydroxide to a faint permanent pink. Keep in a stoppered flask until wanted.

Standardize the barium hydroxide against the hydrochloric acid in the usual manner. Employ some wash water for diluting in place of distilled water, which contains some carbon dioxide.

Measure into the filter bottle from a burette about 13 c.c. (or an amount slightly more than equivalent to the barium hydroxide used) of N/50 hydrochloric acid, the exact amount being obtained from the burette readings.

After the absorption is finished remove the rubber stopper from the bottle, and wash the stopper with a little of the wash

water, letting the washings run into the bottle. Insert the two-hole rubber stopper with connections for filtering and invert as shown in the figure.

Open the upper stop cock and turn on the pump. Now slowly open the filter stop cock and control the flow of liquid entirely with this cock. The barium carbonate remains on the asbestos, and the clear baryta solution which passes through is at once neutralized by the hydrochloric acid. When all the liquid has passed through allow the pump to act for a few minutes until the bottle is partially exhausted, then close the filter cock.

Pour some of the wash water into a small beaker, dip the end of the longer tube into it, and by opening the stop cock allow about 20 c.c. to flow into the bottle before closing it. Unclamp the bottle and shake thoroughly while held horizontally and still attached to the filter. Clamp it in place again, turn on the pump, and draw the wash water through the filter. Repeat this twice. Generally at the third washing the wash water no longer turns pink, showing that the barium hydroxide has been completely removed. If the pink color persists wash again.

Remove the filter bottle and titrate in the bottle, for the excess of acid, with barium hydroxide. The end point is a distinct pink which is permanent for one minute.

To obtain the amount of carbon dioxide subtract the number of cubic centimeters of N/50 acid used from the number of cubic centimeters of acid equivalent to the barium hydroxide used. This will give the amount of carbon dioxide in the sample in terms of N/50 acid, from which the actual number of grams of carbon dioxide can be obtained. From the table in the appendix * obtain the weight of one cubic centimeter of carbon dioxide for the conditions of temperature and pressure observed when the sample was taken. From this the volume of carbon dioxide in the sample can be calculated, and knowing the volume of the bottle, and making allowance for the 25 c.c. of alkali

* Dietrich's Table, the one in general use, is not absolutely correct, the weight of a cubic centimeter of carbon dioxide at 0° C. and 760 mm. being somewhat different from that given at present by the best authorities, but it is sufficiently close for any but the most exacting work.

added, the parts of carbon dioxide per 10,000 of air can be calculated.

A sample calculation follows:

Standardization: — 1 c.c. $Ba(OH)_2$ = 0.48 c.c. N/50 HCl.

Volume of bottle = 991 c.c. Temperature = 18° C. Barometer = 764 mm.

Total $Ba(OH)_2$ used 58.02. HCl used = 26.08.

58.02 c.c. $Ba(OH)_2$ = 58.02 × 0.48 = 27.85 c.c. HCl.

27.85 − 26.08 = 1.77 c.c. N/50 acid equivalent to the CO_2 present.

Since 1 c.c. N/50 acid = 0.44 mg. CO_2, then there are present in the sample 0.78 mg. CO_2.

1 c.c. CO_2 at 18° and 764 mm. weighs 1.817 mg. ∴ 991 − 25 = 966 c.c. of air contains .429 c.c. CO_2 or 4.4 pts. CO_2 per 10,000.

If the amount of carbon dioxide present exceeds 25 parts per 10,000, either a 500 c.c. bottle may be used for collecting the samples, or double the quantities of barium hydroxide and hydrochloric acid should be added. Such a condition rarely exists in practical work.

Pettenkofer Method. — The method which for many years was generally employed for the estimation of carbon dioxide in the air of rooms is a modification of that originally devised by Pettenkofer.* While this method is convenient, and for a long time has been the favorite, it is now quite generally recognized that it contains inherent sources of error which can be obviated only by the use of complicated apparatus and extreme skill in manipulation. It should, therefore, be borne in mind that the results obtained are generally too high even though agreeing closely among themselves.

Principle. — The principle is essentially the same as that of the Walker method, i.e., the absorption of carbon dioxide from a known volume of air in barium hydroxide solution and the titration of the excess with standard sulphuric acid.

* Pettenkofer, *Annalen*, **2**, *Supp. Band*, 1862, p. 1; Gill, *Analyst*, 1892, **17**, p. 184.

The samples are collected in four- or six-liter bottles, as described on page 24, each provided with a rubber stopper carrying a glass tube over which a rubber nipple or cap is slipped. Note particularly the temperature and barometric pressure.

Reagents and Apparatus. — The solutions used are sulphuric acid of such a strength that one c.c. equals one milligram of carbon dioxide (see appendix B), and barium hydroxide solution of approximately equal strength. Since it is impracticable to prepare exact solutions of barium hydroxide, and to keep them without change, the exact value of the barium hydroxide solution must be found by titration against the standard sulphuric acid. This standardization, as well as the subsequent titration, is best made in a small flask to lessen the error from absorption of carbon dioxide from the air. It will be found most generally satisfactory to measure into the flask about 25 c.c. of the barium hydroxide, add a drop of phenolphthalein solution, and titrate with the sulphuric acid to the disappearance of the pink color. In all cases the first end-point should be taken as the correct one, because the pink color will sometimes return on standing.

The apparatus consists of the collecting bottles, 50 c.c. burettes, a stoppered bottle of hard glass of 40 c.c. capacity, and a 25 c.c. pipette.

Procedure. — Remove the cap from the tube in the stopper of the bottle, insert the tip of the burette so that it projects into the bottle, and run in rapidly 50 c.c. of barium hydroxide from the burette. Replace the cap, place the bottle on its side and roll or shake it at frequent intervals for 45 minutes, taking care that the whole surface of the bottle is moistened with the solution each time. At the end of this time thoroughly shake the bottle to mix the solution, remove the cap, and pour the solution into a stoppered bottle of hard glass of 40 c.c. capacity, taking care that the solution shall come in contact with the air as little as possible. Under these conditions a full well-stoppered bottle may safely stand for days before titration. For the titration, measure out with a pipette 25 c.c. of the clear liquid

into a 75 c.c. flask and titrate it with the sulphuric acid as in the standardization.

The calculation is similar to that given under the Walker method except that it should be remembered that only one-half of the barium hydroxide was used in the titration.

Rapid Methods. — In addition to the above methods for determining carbon dioxide just described, there are general tests which can often be used with advantage. If within the space of a few hours some 50 or more tests are to be made, and comparative results rather than great accuracy are required, some simpler form of apparatus is desirable.

Such an apparatus, to be satisfactory, should meet, so far as possible, the following requirements:

(1) It should be sufficiently compact and portable to be carried in the hand from place to place.

(2) It should be as simple in construction as possible, and its use should not involve delicate measurements.

(3) If possible, the apparatus should be made entirely of glass, avoiding prolonged contact of corks or of rubber connectors with any dilute solution which may be used.

(4) It should be so constructed as to protect the solution at all times from the carbon dioxide of the air, especially while the determination is being made, because of necessity such an apparatus must be used within the area of contamination.

(5) The complete apparatus should be sufficient for 50 or more determinations.

(6) It must be capable of giving results of a reasonable degree of accuracy, say within 0.5 part of carbon dioxide in 10,000 parts of air, in the hands of persons having little or no chemical knowledge and minimum skill in manipulation.

(7) If a solution be used in the apparatus it should be one which can be prepared easily from chemicals readily obtained; the solution must maintain its efficiency for a reasonable length of time, if protected from external influences; and the solution should be one that is not at all dangerous or obnoxious to use.

Simplicity of apparatus is much to be desired, but it should

not be gained at too great sacrifice of accuracy. Even when no greater precision is required than is necessary to meet the demands of practical work, it is out of the question to measure the test solution by means of an ordinary pipette or to preserve it for any length of time in stoppered vials; the strength of the solution is almost certain to be reduced by contamination with the breath, or by contact with rubber or cork.

It must ever be borne in mind that extreme care is necessary in the preparation and use of these very dilute solutions, the strict observance of conditions which might well be neglected in ordinary analytical procedures being here an essential factor of success.

For the preservation and measuring of the test solution an apparatus has been devised which appears to answer the above requirements, and in actual practice has been found satisfactory.*

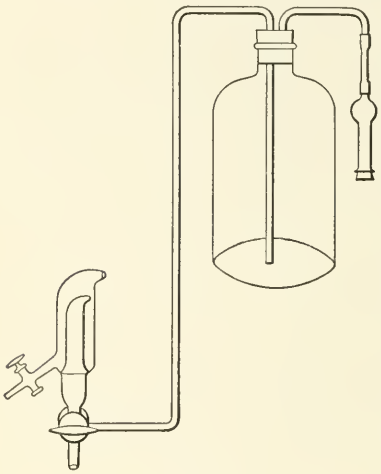


Fig. 5.

The essential feature of this apparatus consists of an automatic pipette for measuring the test solution. This is a modified form of the pipette first proposed by G. P. Vanier and in use in this laboratory for a number of years. A general idea of it may be had from Fig. 5. The manner of using it is extremely simple. The test solution is preserved in a one-liter bottle of hard glass provided with a doubly perforated rubber stopper. Through one opening passes the siphon tube of the pipette, which is sufficiently long to reach to the bottom of the bottle; through the other passes a glass tube ending just below the stopper and connected with a small

* "Air Testing for Engineers," A. G. Woodman and Ellen H. Richards, *Tech. Quar.*, 1901, **14**, p. 94.

drying tube containing fresh soda-lime. By means of the three-way cock the solution is allowed to flow into the small inside pipette until it overflows. The stop cock is then turned, and the solution allowed to flow out at the lowest point. The pipette is made of such a size as to deliver exactly 10 c.c. The excess of liquid which accumulates in the overflow reservoir may be drawn off when desired. The bottle and pipette are contained in a wooden case, about 20 by 8 by 7 inches, outside dimensions, and with the solution, weigh about eight pounds. The case is furnished with a handle at the top so that it may be carried readily in the hand from place to place. The bottle is fastened to the case, and the lower end of the pipette is clamped to a wooden support to keep it from swinging. The stopper should be firmly fastened to prevent loosening.

The bottle should be thoroughly cleaned and washed with potassium bichromate and sulphuric acid, and it is best also to steam it for half an hour or so. As a further measure of precaution the rubber stopper is boiled with dilute caustic potash and thoroughly washed, although the solution can come in contact with it only through splashing while the case is being carried.

This measuring apparatus may be used with a variety of methods, and with various strengths of solution.

Cohen and Appleyard Method.* — *Principle.* — The method of Cohen and Appleyard is based upon the fact that if a dilute solution of lime-water, slightly colored with phenolphthalein, is brought in contact with a sample of air containing more than enough carbon dioxide to combine with all the lime present, the solution will be gradually decolorized, the length of time required depending upon the amount of carbon dioxide present. That is, the quantity of lime-water and the volume of air remaining the same in each case, the rate of decolorization will vary inversely with the amount of carbon dioxide.

Reagents and Apparatus. — The solution used is a dilute solution of lime-water colored with phenolphthalein. To freshly

* *Chem. News*, 1894, 70, p. 111.

slaked lime add 20 times its weight of water in a bottle of such size that it is not more than two-thirds full. Shake the mixture continuously for 20 minutes, and then allow it to settle over night or until perfectly clear. The resulting solution is the stock lime solution, or " saturated lime-water." If made in the manner indicated, each cubic centimeter of it ought to be very nearly equivalent to one milligram of carbon dioxide. If, however, it is desired to know the strength of it more exactly, it may be determined by standard acid.

To prepare the " test solution," pour into the one-liter bottle of the testing apparatus one measured liter of distilled water, and add 2.5 c.c. of a solution of phenolphthalein (made by dissolving 0.7 gram of phenolphthalein in 50 c.c. of alcohol and adding an equal volume of water). Stand the bottle on a sheet of white paper and add the " saturated lime-water " drop by drop from a pipette, shaking the bottle thoroughly after each addition until a faint pink color is produced which is permanent for one minute. Now add 6.3 c.c. of the "saturated lime-water," shake, and immediately connect the bottle again to the apparatus.

For accuracy in air which is high in carbon dioxide, it is found advantageous to use a solution which is twice as strong as the above. This double solution is prepared in precisely the same way, using 5.0 c.c. of the phenolphthalein solution and 12.6 c.c. of the " saturated lime-water."

While this procedure does not give an exact volume of solution, it is believed to be the best for the preparation of this dilute test solution, since it obviates the necessity for pouring the prepared solution from the measuring flask into the bottle in which it is kept; 12.6 c.c. of the stock lime solution is added rather than 10 c.c., in order to keep the values obtained with the resulting solution more nearly comparable with the older values calculated on the supposition that 10 c.c. of the " saturated lime-water " was equivalent to 12.6 mg. of carbon dioxide.

The apparatus used is that shown in Fig. 5. The samples are collected in 500 c.c. bottles by either the water displacement or steam vacuum method.

Procedure. — Remove the rubber stopper from the bottle containing the sample of air; run in quickly by means of the automatic pipette 10 c.c. of the standard test solution; note the time; replace the stopper; shake continuously and vigorously until the pink color disappears; and again note the time. The disappearance of color can most easily be seen if the bottle is held over a piece of white paper. From the time required for the pink color to disappear, the amount of carbon dioxide may be found from Table A.

TABLE A

Time, minutes and seconds.	Standard solution. CO_2 in 10,000.	Double solution. CO_2 in 10,000.	Time, minutes and seconds.	Double solution. CO_2 in 10,000.
0.15	.....	.....	5.45	4.0
0.30	15.6	.....	6.00	.....
0.45	12.1	.....	6.15	3.9
1.00	9.9	16.0	6.30	.....
1.15	8.4	13.1	6.45	3.8
1.30	7.2	11.4	7.00	.....
1.45	6.3	10.1	7.15	.....
2.00	5.5	9.1	7.30	3.7
2.15	4.9	8.3	.....	.....
2.30	4.4	7.6	.....	.....
2.45	4.0	7.0	.....	.....
3.00	3.8	6.5	.....	.....
3.15	3.7	6.1	.....	.....
3.30	3.6	5.7	.....	.....
3.45	.....	5.4	.....	.....
4.00	.....	5.1	.....	.....
4.15	.....	4.9	.....	.....
4.30	.....	4.7	.....	.....
4.45	.....	4.5	.....	.....
5.00	.....	4.3	.....	.....
5.15	.....	4.2	.....	.....
5.30	.....	4.1	.....	.....

Shaker Methods. — At least two forms of apparatus are on the market for determining the percentage of carbon dioxide by measuring the amount of air required to decolorize the standard solutions described on page 38. These are known as the Fitz and the Wolpert Shakers (see Fig. 6). The results obtained are less accurate and more uncertain than by other methods, but if great care is taken to keep the apparatus at some

distance from the face of the worker, approximate results can be obtained. As both shakers operate on the same principle only the Fitz will be described. It consists of a tube of about 30 c.c. capacity, closed at one end, and graduated for a distance of 20 c.c. from the closed end. In this tube, by means of a rubber collar, slides a smaller tube which is contracted at the outer end so as to be more readily closed by the finger.

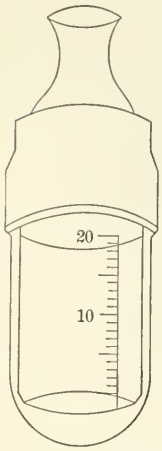


Fig. 6. Fitz Shaker

Procedure. — See that the inner tube of the shaker slides readily in the outer one, moistening the rubber collar slightly if necessary. Have the inner tube pressed down to the bottom of the larger one and measure into the apparatus 10 c.c. of the test solution from the automatic pipette. Pull the inner tube up to the 5 c.c. mark (the bottom of the inner tube serving as the index) and close the end of the tube with the finger. Hold the apparatus horizontally, and shake it vigorously for exactly 30 seconds. The amount of air that is thus brought in contact with the solution is equivalent to approximately 30 c.c., as there are 25 c.c. of air above the liquid when the small tube is forced to bottom of the larger. Remove the finger, press down the small tube again to the bottom of the larger and draw it up to the 20 c.c. mark. Shake the apparatus again for 30 seconds. The amount of air brought in contact with the solution is now 30 + 20 = 50 c.c. Repeat the shaking, using 20 c.c. of fresh air each time, until the pink color is discharged. The amount of carbon dioxide corresponding to the number of cubic centimeters of air used will be found in Table B.

Carbon Monoxide. — The detection and estimation of carbon monoxide in the very minute quantities in which it is found in the air of ordinary rooms is a problem of considerable difficulty.

Detection. — Probably the most convenient test for detecting small quantities is the blood test. Dilute a large drop of human blood, freshly drawn by pricking the finger, to 10 c.c. with water.

Divide the solution into two equal portions, and shake one portion gently for 10 minutes in a bottle containing about 100 c.c. of the air to be tested. Compare the tints of the two portions by holding them against a well-lighted white surface. The presence of carbon monoxide is indicated by the appear-

TABLE B

Cubic centimeters of air.	Standard test solution. CO_2 in 10,000.	Double solution. CO_2 in 10,000.
50	15.6	22.2
70	12.4	18.0
90	10.2	15.1
110	8.7	13.0
130	7.5	11.3
150	6.6	9.9
170	5.8	8.8
190	5.2	8.0
210	4.8	7.3
230	4.5	6.8
250	4.3	6.3
270	4.1	5.9
290	3.95	5.6
310	3.8	5.4
330	3.7	5.1
350	3.6	4.8
370	.....	4.7
390	.....	4.5
410	.....	4.4
450	.....	4.2
490	.....	4.0
530	.....	3.9

ance of a pink tint in the blood which has been shaken with air. One part in 10,000 can be detected in this way.* The delicacy of the test can be increased by examining the blood, after shaking with air, with a spectroscope. By collecting the sample in a eight-liter bottle and examining it in this way 0.01 part in 10,000 may be detected.

Determination. — Practically all the methods for the determination of carbon monoxide in small amounts depend on the equation:

$$I_2O_5 + 5\,CO \rightarrow 5\,CO_2 + I_2;$$

* Clowes, "Detection and Estimation of Inflammable Gas and Vapor in the Air," p. 138.

then either the iodine * is titrated or the carbon dioxide determined. The method consists of passing the air through U-tubes containing potassium hydroxide and sulphuric acid to remove unsaturated hydrocarbons, hydrogen sulphide, etc., and then through a U-tube containing iodine pentoxide, and heated to 150° C. The iodine liberated is absorbed in a solution of potassium iodide, and may be titrated with N/1000 sodium thiosulphate, or the carbon dioxide passing through the potassium iodide may be absorbed by barium hydroxide and determined.†

Nitrites. — The determination of the amount of nitrites or nitrous acid in the air can be readily made as follows: Collect a sample of the air in a calibrated eight-liter bottle, as in the determination of carbon dioxide. Add 100 c.c. of approximately N/50 sodium hydroxide solution. (This should be free from nitrites, and is best made by dissolving metallic sodium in redistilled water.) Shake the bottle occasionally and let it stand for about 24 hours. Take out 50 c.c. of the solution and determine the amount of nitrites as directed in the determination of nitrites in water.

Micro-organisms.‡ — The determination of bacteria in the air is of importance only under special conditions which sometimes exist in dairies, factories, etc. In general the method used is to filter a measured amount of air through sand, shake out the bacteria with sterile water, and plate aliquot portions. Counts are made after 5 days' incubation at 20° C.

* Kinnicutt and Sanford, *J. Am. Chem. Soc.*, 1900, **22**, p. 14.
Morgan and McWhorter, *J. Am. Chem. Soc.*, 1907, **29**, p. 1589.
Seidell, *J. Ind. Eng. Chem.*, 1914, **6**, p. 321.
Gautier, *J. Gas Lighting*, **121**, p. 547.

† For details of the methods reference should be made to the above articles. Recently a portable apparatus has been described by Goutal, *Analyst*, 1910, **35**, p. 130.

‡ See "Standard Methods for the Bacterial Examination of Air," *Am. J. Pub. Health*, 1910, **6**, No. 3, or reprint by the *Am. Pub. Health Assn.*

CHAPTER IV

WATER: ITS RELATION TO HEALTH, ITS SOURCES AND PROPERTIES

TWO-THIRDS of the animal organism consists of water; this water is necessary * for practically all physiological processes, either taking part in the reaction or acting as a solvent. It aids in carrying nourishment to all parts of the body and in disposing of the waste products formed. The evaporation of water from the surface of the body serves as the most important method of regulating the body temperature. Since water is lost by these means as well as during respiration, it is evident that the animal organism must be supplied with water from outside sources. The daily amount needed for each person is five or six pints. This water is derived in part from food which, as eaten, contains from 30 to 95 per cent; in part from boiled water, as in tea and coffee; or raw from well or city tap.

Water is also required for many other purposes, such as cooking, washing, generation of power, and other manufacturing uses. It has been estimated that 25 gallons per person per day is sufficient for household purposes. Then some must be allowed for public use and a rather large amount for manufacturing. For cities in this country amounts varying from 50 to 200 gallons are used, with an average of close to 100 gallons. This is about three times as much as is used in European cities, and undoubtedly a large amount represents unnecessary waste. That this is true is shown by the fact that when the individuals in a community are required to pay for the actual amount of water consumed, which is done through the introduction of meters, the consumption falls off to one-half or one-third of the former quantity used. Waste of water represents a very serious problem in large cities, where it is often necessary to go long

* See "Text-book of Physiological Chemistry," Abderhalden-Hall, John Wiley & Sons, 1908, p. 354.

distances at great expense, to obtain a sufficiently large supply suitable for drinking purposes.

The problem is made still more difficult by the use of large bodies of water, both lakes and rivers, for the purposes of waste disposal. Recent reports of experts * have raised the question as to how much of the expense of purifying a sewage should be borne by the community emptying its waste into a stream, and how much should be borne by a community farther down the stream where water is removed for domestic use. The only certain condition which should be demanded is that wastes should be in such a state and so diluted that no nuisance will be created along the banks of the stream. It seems as if the question of further purification would have to be decided for each individual case as it arises.

That there is a close relation between drinking water and disease has long been suspected, but it is only since the development of the present ideas of the cause of disease that this relationship has been satisfactorily demonstrated. Drinking water may act as the carrier of the germs of at least two well defined diseases, — Asiatic cholera and typhoid fever, — and probably of those of other intestinal troubles. There is, besides, some tendency to disturb the system when a change is made from one kind of drinking water to another of radically different composition, such, for example, as a change from a hard Middle West water to a soft New England water. The disturbance is generally only temporary, as the system becomes rapidly accustomed to new conditions.

The first cholera epidemic to be traced definitely to drinking water was that in London in 1854, which centered about the Broad Street Pump, and the investigation of which was thoroughly carried out by an efficient health officer. Since then numerous epidemics have been traced to the use of polluted water, notably that of Hamburg in 1892–3.†

* See, for example, *Eng. Rec.*, 1912, **65**, p. 209.

† For a description of epidemics of both cholera and typhoid fever see Sedgwick's "Sanitary Science and Public Health."

In this country we have little to fear from cholera on account of the efficient work of the Public Health Service at our ports, but typhoid fever is still a scourge and a disgrace. As early as 1850 it was maintained by Budd in England that this fever was spread by drinking water, but no sufficient evidence was produced until the Lausen, Switzerland, epidemic of 1872. The first large epidemic in this country to be traced to water was that of Plymouth, Pa., in 1885, in which about 1000 cases resulted from the negligence of an attendant on one typhoid patient. Since that time numerous small and large epidemics have been traced with more or less certainty to the use of polluted water.

That the introduction of a good water supply in place of a bad one results in a marked decrease in typhoid fever can be readily seen by almost endless lists of statistics of cities and towns which have either obtained a new supply or have introduced filters, the deaths from typhoid being from one-half to one-fifth of the number formerly recorded in such places.*

Not only does the introduction of unpolluted water mean a decrease in typhoid fever, but there seems also to be a general increase in the health of the community. This effect was noticed at about the same time by Mills in this country and Reincke in Germany, and is known as the Mills-Reincke phenomenon. Hazen attempted to formulate a mathematical relationship between the decrease in typhoid fever and that in all other diseases, but the result is merely an approximation. This increase in the general health may be due to increased vitality by the elimination of one disease. It has been recently suggested that since tuberculosis is liable to follow typhoid fever, a decrease in the latter would account for a decrease in the former.

Safe water, is, therefore, one of the necessary requirements of any community, large or small.

Rain Water. — Let us trace the cycle through which water passes, and point out the sources of supply, and the methods of contamination. Water vapor rising from the sea and land con-

* See *Am. J. Pub. Health*, 1913, 3, p. 1327.

denses and falls to the earth as rain. As it does so, ammonia, carbon dioxide, and other soluble gases are absorbed, and dust and living organisms are collected. As soon as these substances are removed from the air, the rain water becomes a very pure source of supply, and can be used for drinking purposes if properly stored. There are several factors to be observed in this. First, there should be no connection whatever between the storage tank and any drain or sewer from a house or barn. More than one case of typhoid fever has resulted from the backing up of sewage through an overflow pipe into a rain water tank. Second, no metal or other material which is injurious to health should be used in building such a tank, as rain water is soft and often slightly acid and, therefore, has considerable solvent power for most metals. The best materials to use are cement, slate, or stoneware; lead should be absolutely avoided, and zinc will not last any length of time. Third, there should be some method of wasting the first rain that falls, in order not to load the storage tank with dirt and other material which may come from a roof or collecting shed, and render the water unpalatable. Fourth, there should be some easy means of cleaning the tank, and this should be done at frequent intervals. Rain water is used for drinking practically only in tropical regions.

Surface Waters. — Approximately one-third of the rain evaporates again from the surface where it falls; another third runs off on the surface, forming streams, rivers, and lakes, finally reaching the ocean; the other third sinks into the ground, perhaps joining the surface waters underground, coming out as springs or flowing wells, or remaining in the soil. The average rainfall for the whole United States is about 36 inches, varying in different parts of the country from almost nothing to 60 inches. Thus we find the amount of water with which we have to deal is very variable, depending on the locality and the season. Approximately one-half of the rainfall finds its way finally into rivers, either running off on the surface, or entering from underground.

Surface waters form an exceedingly important source of supplies, as most large cities find them necessary on account of the large quantities of water required. Water from small streams and brooks on a water shed may be collected and stored in reservoirs. This method is considerably used, particularly in hilly regions, and if proper care is taken to prevent any pollution on the watershed, sufficient supplies of excellent quality may be obtained. The reservoirs are generally uncovered, and should be stripped of all plant life. Surface water, if unpolluted, usually improves on storage.

Where it is not possible to obtain a supply in this manner, large rivers or lakes are used. These are nearly all subject to more or less pollution, and in general the water should not be used unless filtered or sterilized. Some self-purification will take place in such bodies of water.* The most important factor in such purification is the removal of bacteria by means of sedimentation, the larger particles in the water carrying bacteria with them to the bottom of the stream where the pathogenic varieties soon die out. Thus in a slow moving stream harmful organisms are removed more quickly than in a rapidly moving river. Another important factor is the exhaustion of the food supply. Also, conditions of temperature are not favorable for the growth of many bacteria, and it is undoubtedly true that even in a highly polluted water there is little multiplication, and much dying off of disease organisms.

On the other hand, it is not safe to rely on self-purification, particularly when the health of a large number of people is at stake. There are too many possibilities of accidental pollution. Some artificial means must be used. These will be mentioned later.

Odors sometimes develop in stored water as a result of growth of various plants and animals.† Some of these odors are ex-

* See Jordan, "Natural-purification of Streams." Paper presented at the 26th annual convention of the American Water Works Assn.

† See Whipple, "The Microscopy of Drinking Water," John Wiley & Sons, 1914.

ceedingly disagreeable and may render a water supply unfit to deliver. The growths can generally be exterminated by the proper use of copper sulphate in quantities which will kill the small organisms but are not injurious to the human system (one part to from one to 20 million parts of water).

Surface waters often have color, produced usually by solution, in colloidal form, of partly decomposed vegetable matter, which is perfectly harmless, and such waters should not be condemned unless sewage is also present. They are, however, often decolorized, before delivery, by means of alum. Surface waters are generally softer than ground waters, have a slight, but not disagreeable odor, may be more or less turbid, and in the summer time are liable to be warmer than is desirable. On the whole, however, there is no more satisfactory supply for a large city than a good surface water.

Ground Waters. — From 25 to 40 per cent of the annual rainfall in temperate regions soaks at once into the ground, and passing downward through the soil to hardpan, to clayey or impervious layers, or to rock surface, thence through crevices, broken joints, or glacial drift-deposits to the water-table, flows along the slope for many miles until it finds its way again to the surface, either from the bottom of a lake, the bed of a river, the side of a hill, supplying wells or appearing as springs. In any one of these courses it may be intercepted by man and caught or pumped for his use. Such water may never have been far from the surface; it may have been used and returned to the ground many times; it may have appeared as surface-water and again disappeared to great depths. It has been estimated that water moves in the ground at rates varying from 0.2 to 20 feet per day. This movement is in the form of a sheet, and its rapidity as well as the amount of water held in the ground will depend on the geological formation. Thus a clay will hold more water than loam or sand, while the permeability is just the reverse, clay being nearly impermeable. Water also passes through channels in rocks, either made by the water itself or consisting of cracks and fissures. These latter are often

a source of danger, as no purification can take place if a polluted water travels in this manner.

This long contact with rocks will, of course, bring mineral substances into solution which may be precipitated as new rocks are reached or other streams encountered, so that the same gallon of water may have had many stages in its course, and may have held many different substances in solution. It is no wonder that so active a solvent as water should take with it much substance whenever it remains long in contact with soil or rock, for it may be months before that which has once sunk out of sight again appears. In fact, great rivers are supposed to flow into the sea from under the surface. Then, too, the acquisition of dissolved gases favors the solution of many substances; for instance, water carrying carbon dioxide dissolves limestone.

From a chemical standpoint ground waters may be divided into two classes, — (1) springs and shallow wells (those 30 feet or less in depth) and (2) deep and artesian wells. In general springs and shallow wells yield softer water than deep wells of the same region, but they are much more subject to pollution than the latter, which, if built so as to exclude any surface water, are usually a safe source of supply. Pollution does sometimes enter a deep well, due to the passage of water through fissures and crevices in the rocks.

The greatest source of danger is the shallow well. This should never be used in a thickly populated region, and in country districts only when it can be placed in such a position that there can be no connection through the ground with a privy or cesspool. A well should be built in such a manner that no surface water can enter it, and the walls should be tight to a depth of five to 10 feet below the surface in order that any water which sinks into the ground may be sufficiently filtered before entering the well. The area from which a well may draw varies with the permeability of the soil, and may have a diameter of 20 or more times the depth of the well. The ground which is influenced by a well is in the form of an inverted cone whose apex is at the bottom of the well.

If a well is found upon examination to be polluted with sewage it is often desirable to find the source of trouble in order to stop further pollution. There are several methods of doing this.* A survey of the ground and the conditions surrounding the well is often sufficient to indicate the probable sources, but more definite evidence may be required. Some substance is then added to the suspected source, washed into the ground with a large amount of water, and the well examined for the appearance of the substance. For this purpose bacteria, such as *B. prodigiosus* and *B. violaceous*, can be used. These organisms are easily grown, are harmless and can readily be identified. If these do not reach the well from the suspected source of pollution it is fair to assume that no pathogenic organisms will do so, but will be filtered out in passing through the ground. The only uncertainty with this method is that while the bacteria may be sufficiently removed at the time of the test, the filter may sometime break down and allow sewage organisms, and possibly disease germs, to enter the well. It is, therefore, better not to use a well water which receives sewage from any nearby source, even though bacteria are being eliminated in passing through the ground.

Other methods of tracing the source of pollution are by the use of common salt, easily tested in the well water by an analysis for chloride; lithium or strontium salts, recognized even in minute amounts by means of the spectroscope; and fluorescent dyes such as fluorescein, which are readily observed in a glass of water.

One method of obtaining ground water in comparatively large quantities is by means of the so-called "filter gallery." This consists of a series of wells dug near the banks of a river. It was originally thought that a suction would be created so as to draw water from the river into the wells through a layer of soil sufficient to remove harmful bacteria. As a matter of fact, the filter gallery actually operates by intercepting ground water

* See Thresh, "Examination of Waters and Water Supplies," 2nd edition, pp. 25–34.

on its way to the river, really a better method than had been intended. In sparsely populated regions where the ground water is unpolluted, good results have been and are being obtained by the filter-gallery, but when a region becomes thickly settled considerable danger results. Furthermore, in times of drought water may be drawn from the river bed, and if this reaches the gallery improperly filtered, a typhoid epidemic may result.*

In general, good ground waters contain more mineral matter than surface waters, have no color or odor, can be delivered at a lower temperature, and are often more palatable than surface waters. It is, however, more difficult to obtain large supplies from the ground, and, therefore, only comparatively small communities can avail themselves of such sources.

Water Purification. — Water in passing through the ground may undergo a number of changes in its dissolved and suspended constituents. If this water contains sewage it will carry, with other suspended matter, a large number of bacteria, some of which may be of pathogenic varieties. If the polluted water passes through not too coarse soil, the bacteria will be held by the soil, and thus dangerous disease germs will probably be removed. Even if all the sewage bacteria are not removed there will still be some protection against disease, because disease organisms have, in general, less vitality to withstand unfavorable conditions as well as being present in smaller numbers than less harmful varieties. However, there is still some chance of these bacteria being present at times, and it is, therefore, not advisable to use water in which sewage organisms are present.

In streams, as has already been noted, pathogenic bacteria gradually settle to the bottom and die out.

Thus there is some natural protection against the spread of disease by means of drinking water, but it is not safe to depend on such protection, particularly where the health of a community of people is involved. If a water supply which is sub-

* See "Typhoid Fever in Des Moines, Iowa," *J. Am. Med. Assn.*, 1911, **56**, p. 41.

ject to either continuous or intermittent pollution has to be used, some method of artificial purification is required before it can be safely used for drinking purposes.*

There are two general methods of filtering water on a large scale. The first is known as slow sand filtration. In this method the water is run through a layer of sand from two to four feet thick, supported by gravel and properly underdrained. The filter beds are generally built in units of one acre each, and may be covered or not depending on the climatic conditions. Previous to filtration the water may be screened and stored in reservoirs to allow some removal of suspended matter, including bacteria. As the water passes through the sand a layer of slimy material gradually collects on the surface, which acts as the real straining medium and holds the bacteria. As this material collects the rate of filtration decreases until a point is reached where it is uneconomical to continue. The water is then allowed to drain out from the sand, the top layer scraped off, and the filter again started. The sand removed is washed and returned to the filter about once a year. A slow sand filter operates at rates of from one and a half to three million gallons per acre per day, and is probably the most efficient method of removing bacteria on a large scale. It does not, however, completely remove color or odor.

The other method is that known as rapid filtration (also, unfortunately, termed mechanical filtration). Instead of allowing the filtering layer to form from the matter in the water as in slow sand filtration, a coagulant, generally alum, is added to the water. The alkali, originally present, or added, precipitates aluminum hydroxide which coagulates the suspended particles and removes the color. Part of the hydroxide is allowed to settle out and the remainder is put on a filter built of sand, where it collects on the surface and forms the filtering medium. The filters are washed about every eight hours by reversing the flow of water and agitating the sand by means of rakes or compressed air. Filtration takes place much more rapidly by this

* See Hazen, "The Filtration of Public Water Supplies."

method, being at rates from 100 to 150 million gallons per acre per day. If there is insufficient alkali present naturally in the water enough must be added, usually either as sodium carbonate or as calcium carbonate, to completely precipitate the alum and leave some alkali in excess. Alum, being acid, if allowed to remain in the water renders it corrosive. The amounts of alum used vary from one-tenth to three grains per gallon of water.

Rapid filtration does not give quite as high a bacterial removal as slow filtration, but it is much more efficient in removing turbidity and particularly color. It requires a smaller investment and occupies less ground for the same amount of water filtered. With either method expert control is necessary in order to obtain satisfactory results.

A number of filters on the same principle as just described, but built in small units, are on the market, intended to supply hotels, manufacturing establishments, swimming pools, etc. Many of them give reasonably good results when properly operated, but they never should be considered to be automatic in character. They all need careful attention.

Filters still smaller are sold for office and household uses. These generally consist of artificial stone or porcelain through which the water is forced, such as the Pasteur-Chamberlain or the Berkefeld filter. If the stone, or candle as it is called, is in good condition, sterile water may be drawn when the filter is first put into use, but the bacteria lodging in the stone gradually develop and may grow through the filter so that as water passes through it will wash bacteria with it. It must be admitted that the chances are that pathogenic organisms will not get through. If, however, there is a crack in the candle, often too small a one to be visible, the filter will allow all kinds of bacteria to pass. One of the great objections to the use of such filters is the false feeling of safety which they may inspire in the owners. The all too common small "filter" which screws on the faucet is not only useless, but worse.

If unsafe drinking water must be used in a house, the only

sure method is to bring the water to a boil. This is sufficient to kill any harmful intestinal organisms. Small stills which can be placed on the back of the stove are of service in this connection. The flat taste of boiled water may be removed by the addition of a pinch of salt or by aëration.

Sterilization of Water. — Where a badly polluted supply is used, or extreme caution is desirable, or where a good supply suddenly becomes polluted and emergency measures deemed wise, disinfection may be resorted to. The most practical method is by the use of compounds of chlorine, — hypochlorite of lime (chloride of lime or bleaching powder), sodium hypochlorite (electrolytic bleach), or chlorine gas itself. Of these the cheapest under ordinary conditions is chloride of lime. This has the disadvantage of being disagreable to handle, and of not dissolving completely in water. Amounts of from $\frac{1}{10}$ to $\frac{3}{10}$ grains per gallon are generally sufficient. Sodium hypochlorite can be used where there is cheap electricity, as it is made by passing a current through a solution of common salt. The use of chlorine gas is a recent development and appears to be giving satisfactory results, although considerably more expensive than the other methods.

None of these substances, in the quantities used, are in any way harmful. Where large doses are given complaints are sometimes received that they can be tasted in the water, but, even if true, this is not a necessary consequence of their use. The disinfecting action is probably due to the chlorine itself.

Electrical methods of sterilization are also in somewhat limited use. One of these is through the formation of ozone by an electrical discharge through air, and treatment of the water with the ozonized air. Ozone, in the presence of water, is a reasonably good disinfectant, but its cost makes it prohibitive in most places, and its application to the water presents some engineering difficulty. The largest plant working is probably that at St. Petersburg.*

A more recent development than ozone is the use of ultra-

* See Tillmans-Taylor, "Water Purification and Sewage Disposal."

violet light, as obtained by the quartz-mercury-vapor lamp. Ultraviolet light is a good disinfectant, but it is expensive to produce in most places, and there are difficulties in applying it to waters of all characters. The rays will not penetrate a turbid or colored water to any extent, and, therefore, preliminary filtration and decolorization is often necessary. This, of course, adds greatly to the expense. The method may, however, find use in the future, if it is possible to produce it for a reasonable amount.

Ice. — Questions are often asked concerning the use of ice in drinking water. In general, natural ice, particularly when stored from four to eight months, is comparatively safe. In freezing, suspended and dissolved matter is not removed from the water with the ice, except a small amount mechanically enclosed. Furthermore, it has been shown that over 90 per cent of sewage bacteria die out on storage. Artificial ice, if made from *polluted* water, is not safe, as in the method used all suspended matter is frozen into the center of the cake. If the artificial ice is made from unpolluted or from distilled water as it should be, it is, of course, perfectly safe to use for all purposes.

CHAPTER V

SAFE WATER AND THE INTERPRETATION OF ANALYSES

PURE water, such as may be found in the laboratory, is neither necessary nor probably desirable for drinking. There are, however, certain requirements which should be borne in mind in looking for a supply. First, the water should be free from sewage and all other waste products. Second, it should not contain an excessive amount of mineral matter. Third, it should be free from color, odor, taste and suspended matter, and should be delivered at a temperature not over 15° C. It is obvious that all of these requirements cannot always be lived up to, but it is essential that the first one should be, even at the expense of the other two. A water free from sewage and other waste products can be called a "safe" water. Unfortunately, physical appearance is taken as the criterion of the safety of a supply by too many people. The cool, clear, colorless water is much to be preferred to the safe colored or muddy one; and it is sometimes difficult to persuade the user of such a supply as the former that he may be endangering his health by drinking it when tests have shown the presence of sewage. Since appearance is of such importance, it is necessary to take this into account in any water examination.

Since, as already described, a water once in contact with sewage may become purified and be rendered safe for drinking purposes, and since water is so universally made a carrier of refuse that it is difficult to find a stream or well which has never been at any time in contact with waste, certain arbitrary standards have been chosen to determine when a water may be called safe, on the basis of an analysis. Such limits are very misleading of themselves, especially if used over a wide extent of territory. The English standards, for instance, are not applicable

to eastern North America. Only a study of all local conditions and a wise interpretation of all results can make standard figures of any significance. This is true, also, of bacterial results in surface waters. In lakes and streams there are so many varieties of bacteria present and in such varying numbers, according to wind and rain and water-shed, that taken alone the numerical count gives no more convincing proof than is found in chemical figures.

While it is quite within the limits of possibility that a culture-tube of typhoid bacilli might be emptied into the middle of a river or be washed into a reservoir, and chemical analysis give no sign, yet no continuous natural means of contamination is known which is not accompanied by substances readily detected by suitable chemical examination.

Sanitary Examination. — The examination of a water to determine its safety for domestic use is called a sanitary analysis, in distinction from that examination which determines its fitness for manufacturing purposes, for use in steam boilers, or its medicinal value. Such an examination may be either bacteriological or chemical in character, but the object in either case is the same, that is, to determine the absence of sewage or its presence in quantities sufficient to render the water dangerous to drink. In neither kind of examination are the harmful substances themselves sought for. Typhoid organisms have been isolated from water during epidemics in only a few cases and the process is a long and tedious one. Furthermore, such a search would often be useless for an infected person does not usually come down with the disease until 10 to 14 days after infection, and the organisms might have died during this time. Also, one does not care to wait until an epidemic starts before examining the water supply, but desires to know in advance whether or not there is any possibility of trouble. The presence or absence of sewage determines this possibility.

In a bacteriological examination, the presence of sewage is determined first, by counting the total number of bacteria per cubic centimeter, and second, by looking for some type of dis-

tinctly sewage organism, such as *B. coli*. The total count has little significance in a surface water, but in a well or filtered water, should not be over 100 bacteria per cubic centimeter. *B. coli* should not be present in numbers of one or more per cubic centimeter. Considerable discussion surrounds the determination of this organism, but it is quite impossible to see what difference it makes whether the bacteria isolated show all the typical reactions of *B. coli communis* or not. The members of the colon group get into a water supply practically only with sewage, and it should not make any difference in the interpretation of results, as to what particular member of that group is found. For the methods of making these determinations the reader is referred to some book on bacteriology.*

Before proceeding with the laboratory test of a water, it is essential to know something of the surroundings of the source of supply. So long as the eye can re-enforce the other tests and the whole course of the water may be clearly traced, it is comparatively easy to judge of the character of a supply and of its safety for human use; but when a hole in the ground is the visible source, or the actual history of the water is hidden in unknown distances and depths, the diagnosis is more difficult.

The geological horizon and superficial soil must be studied; the direction and flow of underground water, not the slope of the surface only; the possible sources of danger, occasional as well as constant, within at least a quarter of a mile radius. The composition of unpolluted water of the same region should always be at hand for consultation.

An examination of the environment is often sufficient to condemn a water, but cannot usually give it a clear certificate. Laboratory tests should follow. In the next paragraphs will be found a discussion of the interpretation of sanitary chemical analyses.

Expression of Results. — Results of a sanitary chemical analysis should be expressed in parts of any particular substance

* Prescott and Winslow, "Elements of Water Bacteriology." John Wiley & Sons, New York, 1913.

per million of water. In most cases this is equivalent to milligrams per liter — the exceptions being where the water has an appreciable specific gravity above 1.0, such as sea water.

Accuracy of Methods. — In all water analyses very minute quantities are sought after, and, therefore, all the tests applied must be exceedingly delicate in character. The quantitative results need not, however, be of great percentage accuracy. For example, it makes no particular difference whether 0.050 or 0.055 parts of ammonia per million of water are found — an error of 10 per cent. It might make a good deal of difference if one found 0.2 of a part or 0.05 — a difference of 0.15 parts per million, an amount which in most analytical work would be entirely negligible. The American Public Health Association has suggested that only a limited number of figures be used in reporting an analysis, and thereby eliminate any impression of false accuracy.

Above 10 parts per million. Use no decimals.
From 1 to 10 parts per million. Use 1 decimal.
From 0.1 to 1 part per million. Use 2 decimals.

In the determinations of ammonia and of nitrites 3 decimals may be used.

The above discussion does not mean that the analyses should be made in a careless or slipshod manner, in fact, quite the reverse is true, for there is no kind of chemical work which requires greater care or cleanliness.

As little time as possible should elapse between the collection and examination of samples of water. The more polluted the water the more rapidly will changes take place, and, therefore, all samples should be tested within 24 hours of their collection. Samples for bacterial analysis should be examined immediately, or if sent to a laboratory, should be packed in ice. Sewages and sewage effluents should be analysed within six hours of collection, or if for chemical analysis should be chloroformed (5 c.c. per liter) to prevent chemical changes taking place.

Chemical Examinations. — The chemical analyses generally made in sanitary work are the following: nitrogen as free am-

monia, as albuminoid ammonia, as nitrates, and as nitrites; chlorides in terms of chlorine; oxygen consumed; soap hardness; total solids and loss on ignition; iron; and sometimes oxygen dissolved. The interpretation of the results of each of these will be discussed, and where possible, standard figures will be given.

Nitrogen Cycle. — The most important determinations which must be made in order to decide on the potability of the water in question are those involving the nitrogen compounds and chlorides. A clear understanding of the cycle of nitrogen in nature is, therefore, necessary.

Nitrogen is present in living plants and animals mainly in the form of organic compounds — the proteins and simpler amino compounds. These substances, if boiled with alkaline potassium permanganate, will give off part of the nitrogen in the form of ammonia which can be collected and determined quantitatively. This is called "albuminoid ammonia." When the living plant or animal dies, the proteins are attacked by bacteria and putrefy. In this process the nitrogen is converted first into simpler amino bodies and finally into ammonium salts or substances, such as urea, which readily yield ammonia. Thus, the determinations of ammonia (called "free" ammonia) and of albuminoid ammonia will indicate how far this putrefaction has gone. A waste product, such as sewage, will give, when fresh, both free and albuminoid ammonia in quantity, but on standing, some of the organic nitrogen will change to ammonia, so that the free ammonia will increase and the albuminoid ammonia decrease. Thus, these analyses may be used to indicate fresh or recent sewage pollution of a water supply.

When the organic nitrogen is largely converted to ammonium compounds, and if oxygen is present, another kind of bacteria, called the nitrosomonas, will act on the latter substances and oxidize them to nitrites. This is the second stage in the nitrogen cycle. Thus, the presence of nitrites in a water may indicate less recent pollution than the presence of only free ammonia.

The nitrites, however, are not stable, and if sufficient oxygen

is available, they are oxidized by still another set of micro-organisms, the nitrobacter, giving nitrates. The nitrifying bacteria remained undiscovered for some time, owing to the fact that they do not grow in the laboratory on any medium containing large amounts of organic matter. Thus, the presence of nitrates in a drinking water may indicate contact with sewage at some past time, or as it is called, past pollution.

Nitrates are food for green plants, which in turn die or are eaten by animals, the nitrogen being changed from the inorganic back to the organic form, and the cycle thus completed.

But the cycle is not so simple as would appear. Nitrogen may be lost from it in two ways. While ammonia is being oxidized to nitrites, both may be present and interaction may result with the formation of nitrogen gas.

$$NH_3 + HNO_2 \rightarrow N_2 + 2\,H_2O.$$

Or, nitrites may be reduced by micro-organisms with the liberation of nitrogen. Nitrates may also be reduced to nitrites by bacteria, iron, or possibly by organic matter.

Nitrogen may be added to the cycle as well as lost from it. This takes place by means of the nitrogen-fixing bacteria which occur largely in nodules on the roots of leguminous plants, such as the clover, and also in some soils. These have the power of removing nitrogen from the air and making it available for the plant.

Practical use is made in the septic or Imhoff tanks of the ability of micro-organisms to decompose organic matter and the modern sewage filter is really a culture bed for the development of nitrifying organisms which act on the sewage and render it stable by oxidizing the nitrogen compounds to nitrates.

As will be seen from the above discussion, a sanitary chemical analysis depends primarily upon the determination of the condition of the nitrogen compounds in a sample of water. Each of these will be discussed separately.

Albuminoid Ammonia. — This is the ammonia which is set free by the action of boiling alkaline potassium permanganate

on nitrogenous organic matter. This may have entered the water from perfectly harmless sources, such as dead vegetable substances, or it may have come from waste material, such as sewage. If from the former source, it is relatively stable and, if present in any quantity, is accompanied by color in the water. If from sewage, there may be little or no color, and the nitrogenous matter will be relatively unstable. The stability can be determined by the action of the permanganate, stable substances yielding ammonia only slowly and unstable substances losing it rapidly.

The albuminoid ammonia gives no accurate measure of the total nitrogenous organic matter present, as only about 50 per cent is converted to ammonia, but it does give a good indication of whether or not the organic matter is easily decomposed, and, therefore, whether or not it comes from sewage. A colorless water should not contain over 0.15 parts per million of nitrogen as albuminoid ammonia. The amounts found in good ground waters are generally much lower than this figure. Samples from storage reservoirs, in which there is plant life, may contain larger amounts — up to 0.4 of a part.

The total organic nitrogen as determined by the Kjeldahl method is sometimes used in place of the albuminoid ammonia, but it gives no means of distinguishing between stable and unstable substances, and is not considered in this country to be as good an index of pollution.

Free Ammonia. — This is the ammonia which comes off from a water on direct distillation, the water being made alkaline if necessary. The ammonia is probably present as ammonium salts. Since this represents the first stage in the decomposition of unstable nitrogenous organic matter, its presence in abnormal quantities may be taken as an index of sewage pollution. The amounts of ammonia present in good waters are generally very small, and amounts over 0.15 to 0.2 parts expressed in terms of nitrogen are sufficient to indicate pollution. In general, the free ammonia is less than the albuminoid ammonia. If the reverse is found it is an indication of trouble, unless both are very low.

Cases sometimes arise where abnormally high free ammonia does not indicate sewage, and the analyst should continually be on the lookout for these exceptions. One may be found in wells dug in glacial drift, where ammonia may have come from fossil remains. Another occurs sometimes when a well is located in close proximity to an ammonia refrigerating plant.

Nitrites. — Nitrites in a water are formed either from the oxidation of ammonia or the reduction of nitrates. In either case, they represent an unstable condition, usually accompanied by large numbers of bacteria, and in most cases sewage pollution or surface contamination. As has been said, "a state of change is a state of danger," and the presence of nitrites reveals this condition. As has been mentioned, nitrites may be formed from nitrates by reduction due to iron or organic matter, but such cases are not at all usual.

A good drinking water should be either entirely free from nitrites or should contain them only in very minute quantities. Amounts of 0.01 to 0.02 or more parts per million of nitrogen are sufficient to condemn a water. But while the presence of abnormal amounts of nitrites indicates danger, their absence is no guarantee of the purity of a supply.

Nitrates. — As seen from the discussion of the nitrogen cycle, nitrates are the final stage in the oxidation of nitrogen compounds. Since they are food for plants, we would expect to find only small amounts where there is any plant life. Thus, surface waters are generally low in nitrates while ground waters may be higher. It is probable that practically all nitrates in waters have come originally from animal matter, as vegetable nitrogen is not easily oxidized. In some cases, nitrates have been known to come from chemical fertilizers used on fields.

High nitrates, combined with high chlorides, indicate past pollution. "Past" is used either in the sense of time or distance. That is, fresh sewage may have found its way into a well at some time past, and the nitrogen compounds may have remained there, and been oxidized until, at the time of examination, nitrates predominated over the other forms. Or the sew-

age may have come from such a distance that oxidation has taken place in the passage through the ground.

The presence of high nitrates is not generally accompanied by sewage bacteria, and, therefore, *immediate* danger from the supply does not exist. The objection to using such waters for drinking is, first, that if pollution has once entered, it may enter again, and, second, that the natural filter through which the water is passing may, at some time, fail to work properly and allow sewage bacteria, and with them possibly typhoid organisms, to enter the water. In other words, past pollution indicates a condition of possible future danger, and it is safest to avoid this either by not drinking such water or by watching it carefully by means of frequent examination.

Good surface waters are low in nitrates, over 1 part of nitrogen as nitrate per million of water being a suspicious sign. Ground waters often run much higher than this even when unpolluted, but above 5.0 parts, is in most cases, sufficient to condemn the water as unsafe for drinking.

Chlorides. — In interpreting the results of the analyses of the various nitrogen compounds, it must be remembered that the presence of any one of them in abnormal amounts is rarely sufficient evidence upon which to declare a water unfit to drink. The nitrogen compounds must be accompanied by an abnormal amount of chlorides. Chlorides occur in waters principally as the sodium salt, and as the results of analysis are generally expressed in terms of chlorine, this latter term is the one in common use. Human urine contains about 1 per cent sodium chloride, and the amount of sewage entering a well or stream can be approximately determined by the rise in the chlorine content. Furthermore, chlorine passes through no such cycle as that of nitrogen, and common salt is not taken up by most plants, so that once in a water there is no way by which the chlorine can entirely disappear. If, then, abnormal amounts of chlorine accompanied by abnormal amounts of one of the nitrogen compounds are found in a water, it is a pretty sure indication that sewage, in some state, is entering.

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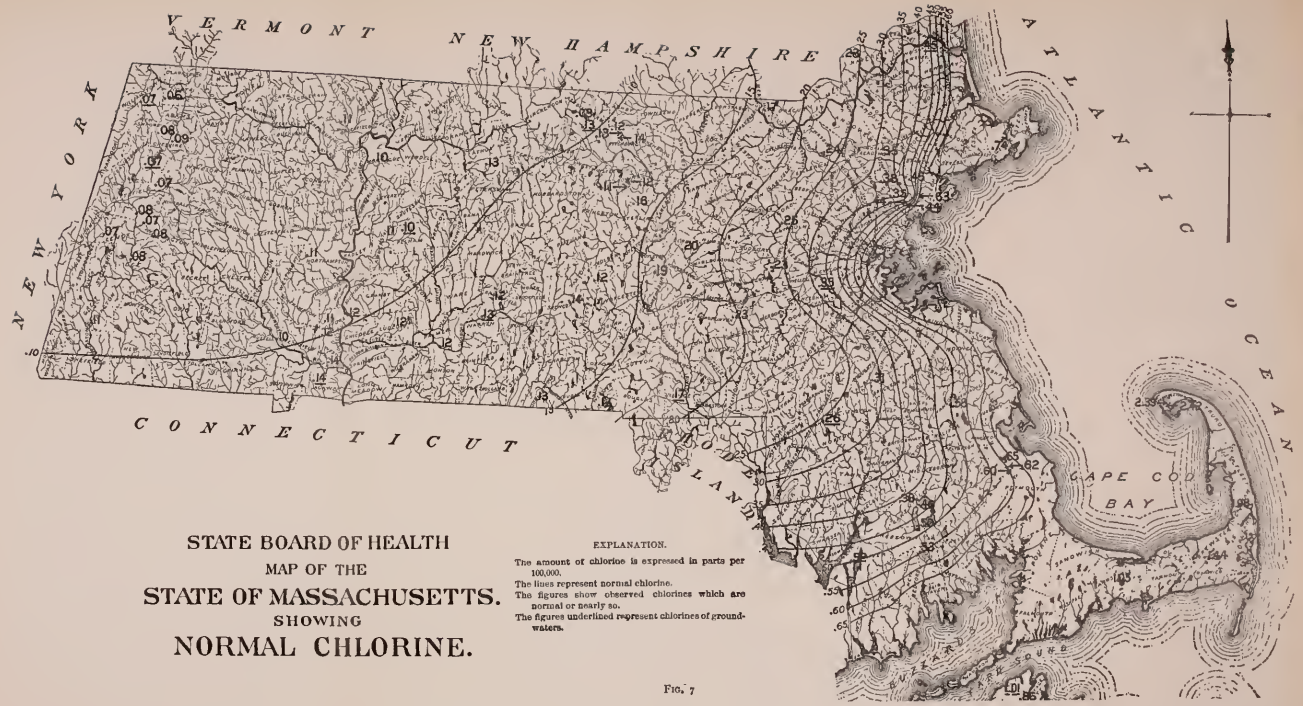


STATE BOARD OF HEALTH

MAP OF THE

STATE OF MASSACHUSETTS.

SHOWING

NORMAL CHLORINE.

EXPLANATION.

The amount of chlorine is expressed in parts per 100,000.

The lines represent normal chlorine.

The figures show observed chlorines which are normal or nearly so.

The figures underlined represent chlorines of ground-waters.

FIG. 7

The difficulty is to decide on what constitutes an abnormal amount of chlorine, since salt occurs, to some extent, in most soils and rocks, and in some places in very large quantities. Some years ago, the Massachusetts State Board of Health attempted to solve this problem by a careful study of a large number of waters from all over the state. The chlorine was determined in those which, from the surroundings and the other constituents, could safely be regarded as free from pollution. The figures obtained were placed on a map of the state at the appropriate places and lines drawn through equal values. These lines were termed "isochlors." This map is shown opposite. (Note. The figures are given on the map in parts per 100,000.) Since this map was made for Massachusetts, a number of other states have made similar ones. The maps give with reasonable accuracy the normal chlorine values for surface waters, but for deep or artesian wells, the figures do not necessarily hold. Consequently, in some states, for example in Illinois, it has been found more satisfactory to give normal values according to the source of the supply.

But the presence of chlorine in amounts above normal, alone, is not sufficient to condemn a water. High chlorine and low nitrogen are sometimes found together in a well water which has been contaminated with wastes from a sink drain. The ratio of nitrogen to chlorine can sometimes be used to distinguish between barn and human sewage, as the former contains less chlorine than the latter for the same amount of nitrogen. Excessively high nitrates with chlorine only slightly above the normal sometimes indicates washings from a fertilized field.

Mineral Matter. — Since water is a universal solvent, it is not surprising to find considerable amounts of mineral matter under the headings "total solids" and "hardness." How much calcium sulphate or magnesium chloride or other soluble mineral matter is allowable in a potable water is for the physician rather than the chemist to say, but it seems to be the consensus of opinion that, for the normal healthy person, the presence of

mineral matter, even in considerable quantities, is in no way deleterious to the system.

As has been said, the human system possesses great adaptability, not only for different foods, but for mineral substances water-carried. Not so the steam-boiler or the laundry-tub, which reacts very sensitively and affects the pockets of the consumers. The determination of sulphates gives an indication as to how the hardness is divided, as permanent hardness is caused principally by calcium sulphate.

In a region of soft water, high solids with chlorine and nitrates indicate sewage pollution. Silica is much more commonly present, even in surface-waters, than is often supposed. What its effect may be is unknown. Iron is not uncommonly found in combination with organic matter in either surface or imperfectly filtered waters in contact with soils poor in calcium salts. It is frequently accompanied by free ammonia, which causes an abundant growth of *Crenothrix*. It is also present in deep wells in the form of bicarbonate, which precipitates on exposure to warm air.

Organic Matter. — The amount of carbonaceous matter, determined either by the oxygen-consumed test or by the loss on igniting the solids, is of little use in interpreting a water analysis; it is too difficult to get concordant results. The latter test may sometimes be of service in a qualitative way, because the residue from a recently polluted water often gives a distinctive disagreeable odor when ignited. In some laboratories, the quantitative determination is omitted entirely.

Dissolved Oxygen. — During the last few years, the determination of the oxygen dissolved in water has assumed considerable importance, because of the use of the test as an indication of the sanitary condition of a harbor or river. As long as sufficient oxygen is present, the putrefactive changes which give off disagreeable odors will not take place. There is some difference of opinion as to how low the oxygen content may be allowed to fall and still prevent these changes, but 40 per cent of saturation is a safe figure to use.

The test is also used to determine the putrescibility of a sewage effluent as described later under that test. The object is to determine the amount of oxygen absorbed by the organic matter in the effluent.

Physical Tests. — These are of little importance as far as the determination of pollution is concerned, but are generally included in an examination in order to satisfy those who insist that a water shall be attractive as well as safe.

Sewage Analysis. — Sewages may be tested to determine their strength and constituents in order to help in deciding upon the best method of treatment, and also as a basis for determining the amount of purification which any process gives. The analysis of effluents is carried on also for this latter purpose, and in order to determine their putrescibility. For an extended discussion the reader is referred to another book.*

Value of Tests. — It is often asked if some tests cannot be made by the ordinary person of average intelligence which will enable him to tell the quality of a water as well as the expert to whom he pays ten or twenty dollars for an opinion. A careful perusal of the preceding pages will have answered the question in the negative. There is no assay of water as there is of gold and silver. Not one, but ten or twenty tests must be made. Not only must the tests be made with the utmost care and cleanliness of person, utensils, and room, but the results must be studied in the light of other experience and other knowledge, geological and biological, and after all this is done, there is an array of circumstantial evidence which must be carefully weighed by one whose judgment and experience enable him to read clearly where another might see nothing. The value of a water-analysis is in direct proportion to the knowledge and experience of the one who interprets it. Clinical skill in addition to theoretical knowledge is as much required to interpret the figures obtained in the course of a water-analysis, as in the diagnosis of a disease; and the analogy goes still further, for just as some diseases are clearly defined, and others

* Fowler, "Sewage Works Analysis."

are so complicated that only those who have had long experience can outline a safe course of treatment, so some waters bear the marks of their character so plainly as not to admit of mistake, while others require most careful study. For these reasons, the value of water-analysis should not be decried because the fears aroused by reports given by unskilled analysts prove groundless, any more than the practice of medicine should be discarded because inexperienced men make mistakes.

Is the water in any given case safe for drinking? To answer this question there is needed a knowledge, wider than a chemist's, of the relation of decaying organic matter and of the germ-carrying power of water to outbreaks of disease. There must be added the knowledge of the biologist, the engineer, and the sanitarian.

CHAPTER VI

WATER: ANALYTICAL METHODS *

WATER-ANALYSIS cannot be carried on in an ordinary laboratory. In order to obtain satisfactory results, it is necessary to have a room set apart for the purpose, and to exclude rigidly all operations which tend to the production of fumes or dust. Where such minute traces of substances are dealt with as in water-analysis, too much care cannot be taken to insure the absolute cleanliness of the apparatus and the surroundings. It is desirable that the room be well lighted, and, if possible, the windows should face toward the north.

For the collection of water samples, glass-stoppered bottles of about a gallon capacity are best. Those used in this laboratory are of white glass, 15 inches high to the top of the stopper, five and a half inches in diameter, and weigh about three pounds. They have flat, mushroom stoppers, on each of which is engraved a number to correspond with that on the bottle. The bottles, before being sent out, are thoroughly cleaned with potassium bichromate and sulphuric acid, washed with distilled water and dried. If glass-stoppered bottles are not at hand, new demijohns fitted with *new* corks may be used. A glass bottle or a demijohn is much to be preferred to an earthenware jug, because, if for no other reason, it is so much easier to be sure that the interior is clean. It should always be borne in mind that in water-analysis the question is one of very minute quantities of material, and that the methods to be employed are extremely delicate. Hence, in the case of many waters, careless handling of the sample would contaminate the water to a sufficient extent to render valueless the results obtained in the laboratory.

* See "Standard Methods for the Examination of Water and Sewage," American Public Health Association, 1912.

In collecting samples, the following directions should be closely followed:*

Directions for Collecting Samples for Analysis. — *From a Water-tap.* — Let the water run freely from the tap for a few minutes before collecting the sample. Then place the bottle directly under the tap and rinse it out with the water three times, pouring out the water completely each time. Place it again under the tap; fill it to overflowing and pour out a small quantity so that there shall be left an air-space under the stopper of about an inch. Rinse off the stopper with flowing water; put it into the bottle while still wet and secure it by tying over it a clean piece of cotton cloth. Seal the ends of the string on the top of the stopper. Under no circumstances touch the inside of the neck of the bottle or the stem of the stopper with the hand, or wipe it with a cloth.

From a Stream, Pond, or Reservoir. — Rinse the bottle and stopper with the water, if this can be done without stirring up the sediment on the bottom. Then sink the bottle, with the stopper in place, entirely beneath the surface of the water and take out the stopper at a distance of twelve inches or more below the surface. When the bottle is full replace the stopper, below the surface if possible, and secure it as directed above. It will be found convenient, in taking samples in this way, to have the bottle weighted so that it will sink below the surface, and to remove the stopper with a cord. It is important that the sample should be obtained free from the sediment at the bottom of a stream and from the scum on the surface. If a stream should not be deep enough to admit of this method of taking a sample, dip up the water with an absolutely clean vessel and pour it into the bottle after the latter has been rinsed.

The sample of water should be collected immediately before shipping by express, so that as little time as possible shall intervene between the collection of the sample and its examination. All possible information should be furnished concerning the source of the water and of possible sources of contamination.

* *Ann. Rept. Mass. State Board of Health*, 1890, p. 520.

For example, in the case of a well, the proximity of dwellings, cesspools, or drains should be recorded, and the character and slope of the soil, whether toward or away from the well, should be noted. In the case of a surface-water, mention any abnormal or unusual conditions; as, for instance, if the streams or ponds are swollen by recent heavy rains, or are unusually low in consequence of prolonged drought, or if there be a great deal of vegetable growth in or on the surface of the water. Record, in short, any circumstantial evidence which by any possibility may aid in the final judgment.

The question of proper collection of samples is an important one, and the chemist is perfectly justified in refusing to give an opinion in regard to the purity of a water which he has not himself collected.

Preparation for Analysis. — Since changes in the composition of a contaminated water are constantly going on, the analysis of the sample should be begun without delay. The bottle is held under the tap, and the neck and stopper are washed free from adhering dust. The stopper is rinsed off with some of the water from the bottle.

If the sample has stood for several hours, allowing suspended matter to settle, the conditions of turbidity and sediment, as described on page 108, may first be observed. The sample is then thoroughly mixed and qualitative tests made for alkalinity, ammonia and chlorides. Make the alkalinity test with methyl orange indicator. If a sample is acid, it is necessary to make alkaline, as described later, before starting the determinations for free ammonia and chlorides. Make the test for ammonia by adding two c.c. of Nessler reagent to 50 c.c. of the sample in a Nessler tube. A reddish-brown color or precipitate means the presence of large amounts of ammonia, and care should be taken not to take too much of the sample for the quantitative determination (see page 74). A qualitative test for chlorides will determine the amount of water to be taken for the analysis, — a very slight opalescence meaning low chlorides, which will necessitate the use of a 250-c.c. sample, while a distinct turbidity or a precipitate will allow a 25-c.c. sample to be used.

As the nitrogen compounds are more subject to important changes than any others, it is desirable to make these determinations first, the order of the remainder being immaterial.

It is essential that the sample of water be thoroughly mixed each time any is withdrawn, as only in this way will the samples removed be of constant composition. This is particularly important in dealing with sewages and sewage effluents, or where there is a considerable amount of suspended matter.

The methods for preparing standard solutions and other special reagents will be found in Appendix B.

Determinations of Free and Albuminoid Ammonia. — Ammonia occurs in waters as ammonium salts, — carbonate, chloride, or nitrate. In sewages it may be partially present as the hydroxide. On boiling an alkaline solution of these substances, the salts are decomposed, as well as some unstable organic compounds such as urea, and ammonia passes off and dissolves in the condensed steam. The ammonia thus collected is called the "free ammonia." If, now, alkaline potassium permanganate is added to the water left after the free ammonia has been removed, and the boiling continued, part of the nitrogenous organic matter will be decomposed with the liberation of ammonia. This is termed "albuminoid ammonia."

The principles involved in the two determinations have been described in the above definitions, that is, the water is first boiled, and the steam condensed until all the free ammonia has been removed. Then alkaline potassium permanganate is added, and distillation continued until no more albuminoid ammonia is evolved. The ammonia is determined in the distillates by means of Nessler reagent which gives a greenish yellow with very small amounts of ammonia, and yellow to reddish brown with larger quantities. The exact amount of ammonia is obtained by comparison of the colors obtained with those from known amounts of ammonia.

Nessler reagent is a solution of potassium mercuric iodide (K_2HgI_4) containing potassium hydroxide. The colored substance formed when this reacts with ammonia is dimercuram-

monium iodide ($NHg_2I \cdot H_2O$), which is an ammonium iodide in which the hydrogen atoms have been substituted by mercury. This substance is slightly soluble in an excess of potassium iodide and potassium hydroxide, giving a color proportional to the amount of ammonia present.

Apparatus and Reagents. — The apparatus consists of a 750 c.c. round-bottomed flask, having square shoulders and a narrow neck five inches long, and an ordinary Liebig con-

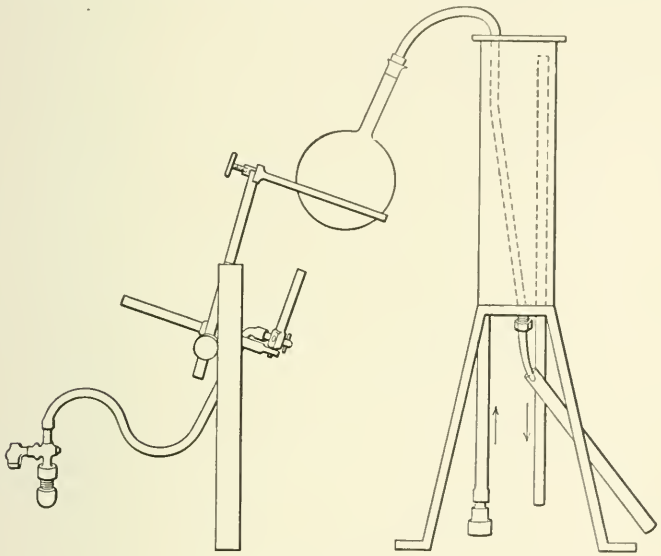


Fig. 8.

denser fitted with a block-tin inner tube $\frac{3}{16}$ of an inch in diameter which extends just through a cork stopper closing the flask. The apparatus is set so that the distillate may be collected directly in a 50 c.c. Nessler tube. The flasks are heated either with the free flame of a Bunsen burner or with an electric flask heater. These latter are somewhat slow in heating up and in cooling, but give an even heat with just about the proper rate of distillation and show little tendency to cause "bumping." In place of the Liebig condenser, the tin tube may be passed through a copper or galvanized iron tank (see Fig. 8),

fitted with proper inlets and outlets, and serving as a condenser for a number of flasks. New flasks are treated with boiling dilute sulphuric acid and potassium bichromate before they are used. New corks should be steamed out for one or two hours. A good sound cork will last for several months with daily use.

The Nessler tubes used should be of the same height up to the 50 c.c. mark.

Reagents necessary are Nessler solution, alkaline potassium permanganate, a standard ammonium chloride solution and ammonia-free water (see Appendix B).

Procedure. — Free the apparatus from ammonia by placing 500 c.c. of ammonia-free water in the flask and distilling. Collect the distillate in 50 c.c. Nessler tubes, and test each tube as it is filled, by adding two c.c. of Nessler reagent and comparing the color obtained after waiting five minutes with that obtained by adding two c.c. of Nessler reagent to 50 c.c. of ammonia-free water. This latter gives a zero standard. Continue until the distillate is free from ammonia and then pour the water left in the flask into the bottle marked " ammonia-free residues."

While this is going on make a qualitative test on the sample of water to determine the amount which should be used for the quantitative determination. To do this, add to 100 c.c. of the water, removed from the bottle only after thorough mixing, one c.c. of 10 per cent copper sulphate solution, and one c.c. of 50 per cent potassium hydroxide. Allow to settle and filter through a dry paper into a 50 c.c. Nessler tube, discarding the first 10 c.c. of filtrate. Add two c.c. of Nessler reagent, and allow to stand for 10 minutes. Make a standard by placing two c.c. of the standard ammonium chloride solution in a Nessler tube, making up to 50 c.c. with ammonia-free water, mixing thoroughly and adding two c.c. of Nessler reagent. If the color obtained from the sample of water is less than this standard, use a 500 c.c. sample of the water for the determination; if equal to or greater than the standard, use a 100 c.c. sample; if the color is so deep that a precipitate forms, use a 10-c.c.

sample. For sewages five or 10 c.c. are sufficient. In case less than 500 c.c. are used, dilute the amount to this volume with ammonia-free water.

Test some of the water with methyl orange for acidity. If acid, 0.5 gram of pure sodium carbonate must be added before starting the distillation. The great majority of drinking waters are alkaline, but once in a while an acid water turns up, and it is well to be on the lookout. Acid sewages and sewage filter effluents are not uncommon.

When the apparatus has been freed from ammonia, shake thoroughly the bottle containing the water sample, and measure out in a calibrated flask 500 c.c., or a smaller amount, according to the qualitative test described above, adding enough ammonia-free water to make the total volume at least 500 c.c., and pour into the distilling flask. If necessary, add sodium carbonate. Distill three portions of 50 c.c. each into well-rinsed Nessler tubes. Regulate the height of the flame so that the time of distilling 50 c.c. shall not be more than eight and not less than five minutes. In most cases three portions are sufficient to collect all the free ammonia, but it is well to test the last portion with Nessler reagent, and compare it with a zero ammonia standard, before proceeding further. Save these portions for nesslerization, as they contain all the free ammonia.

After the free ammonia has been distilled off, allow the contents of the flask to cool for 10 minutes; then add 40 c.c. of alkaline permanganate through a funnel, taking care that none of the alkaline solution touches the neck of the flask, and proceed with the distillation of the albuminoid ammonia. With colored waters distill off five portions of 50 c.c. each; with colorless waters, three or four portions will suffice. These portions contain the albuminoid ammonia.

Unless permanent standards are used, prepare standards by adding to Nessler tubes nearly filled with ammonia-free water varying quantities of the standard ammonium chloride solution; for instance, 0.1, 0.3, 0.5, 0.7, 1.0, 1.3, 1.5, 2.0, 2.5, 4.0, 6.0 c.c. The standard ammonium chloride solution contains 0.00001 gram

N in one cubic centimeter. Mix the contents of the tubes by rotating them between the palms of the hands or by pouring into another Nessler tube and back again (never shake them like a test-tube or stir them with a rod), allow them to stand for a few minutes and add two c.c. of Nessler reagent to each tube and to each of the portions of distillate. At the end of 10 minutes match the colors and record the amount of ammonia in terms of cubic centimeters of the standard ammonium chloride solution. From the value of this solution calculate the amounts of free and albuminoid ammonia as parts of nitrogen per million of water.

As an example, the following results from distilling 500 c.c. may be given.

Free ammonia.		Albuminoid Ammonia.	
1st 50 c.c.,	0.7 c.c.	1st 50 c.c.,	4.5 c.c.
2nd 50 c.c.,	0.3 c.c.	2nd 50 c.c.,	2.8 c.c.
3d 50 c.c.,	0.0 c.c.	3d 50 c.c.,	1.5 c.c.
		4th 50 c.c.,	1.0 c.c.
		5th 50 c.c.,	0.5 c.c.
	1.0 c.c.		10.3 c.c.

In this case, the free ammonia would be 0.020 and the albuminoid ammonia 0.206 parts per million.

In dealing with sewages or sewage effluents, which are very high in free ammonia, if the ammonia were collected in three portions, so much would distill over in the first portion that the color given with the Nessler reagent would often be too deep to read or a precipitate might form. To avoid this, the total distillate of 150 to 175 c.c. is collected in a 200-c.c. graduated flask, made up to the mark, thoroughly mixed by pouring into a clean dry beaker and back again, and then 50 c.c. of it taken for nesslerization. In this way, the ammonia is distributed more evenly in the distillate and the determination is not sacrificed.

If free ammonia only is desired in a sewage or sewage effluent, a direct determination is to be preferred over distillation. For

this, proceed as directed in the qualitative test for ammonia, except that a smaller amount of the filtrate should be used, two or five c.c., and this made up to 50 c.c. with ammonia-free water, treated with Nessler reagent, and matched against standards as just described.

Notes. — Where a large number of determinations are made at frequent intervals, permanent Nessler standards are a great convenience. These should be made according to directions found in "Standard Methods,"* but should be adjusted by comparison with nesslerized standards made from ammonium chloride solution.

It is impossible to convert all of the organic nitrogen into ammonia by boiling with alkaline permanganate. The amount of ammonia which is thus obtained depends not only upon the character of the substances, but also upon the concentration of the solution and the rate of boiling. In order that the albuminoid ammonia in potable waters shall bear some definite relation to the total organic nitrogen, it is necessary that conditions shall be duplicated as nearly as possible in different determinations; that is, the alkaline permanganate must be added to a definite volume of water, and the boiling must be carried on at a definite rate. Some of the highly-colored surface-waters give up their nitrogen very slowly by this treatment; polluted waters, on the other hand, yield the ammonia more rapidly, so that the observation of the relative amounts found in the successive portions is of the utmost importance in forming a judgment.

A depth of color given by six c.c. of the standard ammonium chloride with the Nessler reagent is about the limit of satisfactory comparison in the 11-inch 50 c.c. tubes. The color given by 10 or 12 c.c. of the standard may be matched in the 100 c.c. tubes with a depth of five inches and a diameter of $1\frac{1}{4}$ inches.

For most cases, where great exactness is not essential, it is possible to divide the 50 c.c. or the 100 c.c. portion into two

* "Standard Methods of Water Analysis," American Public Health Association, 1912, p. 17.

equal parts by pouring into a tube the exact counterpart of the standard tube and matching the color. It is even possible to approximate closely the correct result by the use of a foot rule. The standard is, we will assume, five c.c. The height of the liquid in the tube to be tested we will call nine inches. If the height of the column left which matches five c.c. is three inches, then the reading was 15 c.c. of the standard.

The limit of solubility of the mercur-ammonium iodide is reached at 25 or 30 c.c. of the standard in 50 c.c. The incipient precipitate not only changes the color of the solution, but causes a slight milkiness or turbidity which prevents a sharp reading of the color.

The test is an excellent example of quantitative color work when carried out under strictly comparable conditions.

It should, perhaps, be stated that in both the ammonium and nitrate determinations, as also in that of iron, dilution of the sample in which the color is already developed does not give a correct result. Therefore dilution, if necessary, must be made before the reagents are added.

In order to secure the most accurate results, it is important that the temperature of the distillates to be nesslerized and of the standards be the same, since the warmer solutions give a more intense color with the Nessler reagent.

Total Organic Nitrogen, Kjeldahl Process. — The principles involved in the method consist in the oxidation of the carbon and hydrogen of the organic matter with boiling sulphuric acid, the nitrogen being converted into ammonia and held by the acid as ammonium sulphate. The ammonia is then liberated and distilled off from an alkaline solution.

Apparatus and Reagents. — The apparatus used is that shown in Fig. 9. This is an arrangement for distilling with steam. The reagents needed are nitrogen-free sulphuric acid and potassium hydroxide (see Appendix B).

Procedure. — Measure 500 c.c. of the water into a round-bottomed flask of 750 c.c. capacity and boil until about 200 c.c. have been driven off. (The free ammonia which is thus ex-

pelled may be determined, if desired, by connecting the flask with a condenser.) Allow the water remaining in the flask to cool, and add 10 c.c. of pure concentrated sulphuric acid free from nitrogen. Mix by shaking; place the flask in an inclined position on wire gauze under the hood and boil cautiously until the water is all driven off. Place a small funnel in the neck of the flask to prevent the escape of acid fumes, and continue the heating for at least half an hour after the sulphuric acid becomes white. Meanwhile, rinse out the distilling apparatus and free it from ammonia as usual. Then, after the acid in the digestion flask has cooled, rinse down the neck of the flask with 100 c.c. of ammonia-free water and attach the flask to the distillation apparatus. Add 100 c.c. of potassium hydroxide solution through the separatory funnel and distill off the ammonia with steam, receiving the distillate in a 250-c.c. graduated flask. Conduct the distillation rather slowly until the first 50 c.c. have distilled over, then distill more rapidly until about 175 c.c. have been collected. Make the volume of the distillate up to 250 c.c. with ammonia-free water, mix it thoroughly and take 50 c.c. for nesslerization.

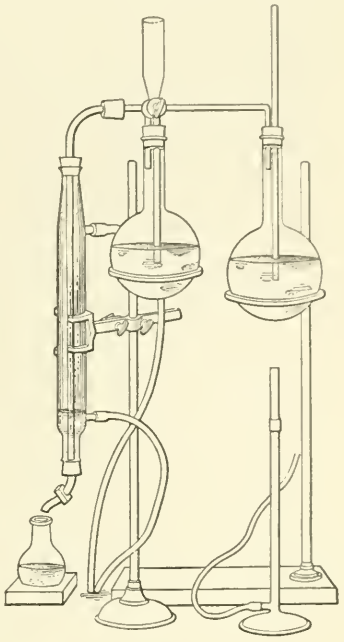


Fig. 9.

The use of mercury and of potassium permanganate to assist in the oxidation has been found to be unnecessary, as the organic matter in natural waters is much more easily oxidized than in other substances, — flour, for instance. The presence of nitrates and nitrites in waters has not been found to interfere with the accurate determination of the organic nitrogen. The error,

which has been found by Kjeldahl and Warrington to be caused by the presence of nitrates seems to disappear when the organic material is diluted to the considerable extent that exists in natural waters. The high chlorine found in some well-waters does not interfere with the method to any extent, but this determination does not possess much value in this class of waters, which are low in organic nitrogen.

In carrying out the digestion with sulphuric acid, the greatest care must be taken to prevent access of ammonia or dust from any source. The acid solutions will absorb ammonia from the air or from the dust of the laboratory if they are allowed to remain uncovered for any length of time. This source of error may in some instances be sufficiently large to render a determination valueless, even in a room which is, to all appearances, free from ammonia-fumes. Hence, the operation should, if possible, be carried to completion within twenty-four hours, and for every set of determinations a blank analysis should be made with ammonia-free water in order to make a correction for the ammonia in the reagents, and for that accidentally introduced during the process.

As the result of many hundred comparative determinations of the organic nitrogen and of the albuminoid ammonia in natural waters which take their origin in the glacial drift, it has been found that the nitrogen given by the albuminoid-ammonia process, as directed in the previous pages, is about one-half of the total organic nitrogen as given by the Kjeldahl process; in the case of sewages and polluted waters, it is very variable owing to their irregular composition.

Determination of Nitrogen in the Form of Nitrites.—This determination depends on the formation of a pink azo dye by the interaction of sulphanilic acid, naphthylamine acetate, and nitrous acid. If an excess of the first two reagents is used, the amount of dye and, therefore, the depth of color will be proportional to the amount of nitrite present in the water. The color is then compared with a series of standards made from a sodium nitrite solution of known strength and the nitrite computed in terms of nitrogen.

The reactions which take place are, first, the diazotizing of the sulphanilic acid by the nitrous acid present, and then the interaction of this diazo compound with naphthylamine to form the colored substance, α-naphthylamine-para-azo-benzene-para-sulphonic acid.

$$C_6H_4\begin{cases}NH_2\\SO_3H\end{cases} + C_{10}H_7NH_2 + HNO_2 \rightarrow$$

$$C_{10}H_6\begin{cases}N=N-C_6H_4-SO_3H\\NH_2\end{cases} + 2\,H_2O.$$

Apparatus and Reagents. — The only special apparatus needed is a number of 100 c.c. Nessler tubes. The reagents used are a standard sodium nitrite solution (1 c.c. contains 0.0000001 gram nitrogen), a solution of sulphanilic acid in acetic acid, a solution of naphthylamine acetate, and a suspension of aluminum hydroxide (see Appendix B).

Procedure. — If the water is colorless, measure out 100 c.c. into a 100-c.c. Nessler tube. If the water possesses color which cannot be removed by simple filtration, it should be decolorized as follows: Thoroughly rinse with the water a 250-c.c. glass-stoppered bottle; pour into it about 200 c.c. of the sample, add about three c.c. of milk of alumina and shake the bottle vigorously. Let stand for 10 or 15 minutes and filter through a small plaited filter which has been thoroughly washed with water free from nitrites. To 100 c.c. of the filtered sample or of the originally colorless water add 10 c.c. of the sulphanilic acid in acetic acid and 10 c.c. of naphthylamine acetate solution. A pink color shows the presence of nitrite. To determine the amount* of nitrite present make up standards by placing 5 c.c., 10 c.c., 15 c.c., and 20 c.c. each of the standard nitrite solution in 100-c.c. Nessler tubes. Make up to 100 c.c. with nitrite-free water, mix by pouring into a Nessler tube and back to the original tube, and then add the reagents as before. Allow to stand 10

* Standard color papers and also acid solutions of fuchsine are used for nitrite standards. Neither of these has been found very satisfactory in this laboratory.

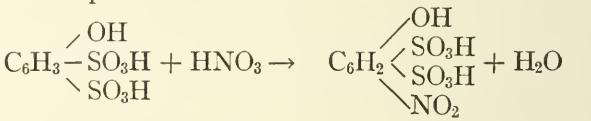
minutes, and match with the color obtained from the water sample. If this does not match any of the standard colors, make up intermediate standards. Do not attempt to match colors closer than to one c.c. of the nitrite solution. If the color is deeper than that given by 20 c.c. of the standard nitrite solution, start a new determination using a smaller quantity of water and diluting to 100 c.c. with the ammonia-free water.

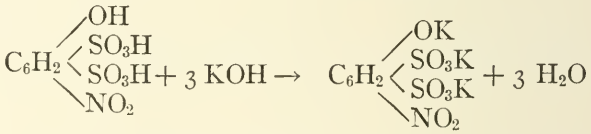
One c.c. of the standard nitrite solution equals 0.0000001 gram nitrogen. Determine the number of c.c. needed to match the color obtained from the water sample and calculate the results in parts of nitrogen per million of water.

Notes. — In case the color obtained is deeper than 20 c.c. of the standard, an aliquot part may be measured, as described under the ammonia determination. This will be sufficiently accurate for most purposes.

When once obtained, the color will remain unchanged for one-half to three-quarters of an hour. If left for a longer time, the nitrites absorbed from the air will noticeably increase the color.

Determination of Nitrogen in the Form of Nitrates.* — This determination depends on the action of nitric acid on phenol-disulphonic to form nitrophenoldisulphonic acid, which gives an intensely yellow color in alkaline solution. The reactions involved can be expressed as follows:





Reagents. — The reagents needed are a standard nitrate solution of which one c.c. contains 0.000001 gram nitrogen, and phe-

* Sprengel, *Fogg, Ann.*, 1863, **121**, p. 188; Grandval and Lajoux, *Compt. rend.*, 1865, **101**, p. 62; Gill, *J. Am. Chem. Soc.*, 1894, **16**, p. 122; Chamot & Pratt, *J. Am. Chem. Soc.*, 1909, **31**, p. 922; 1910, **32**, p. 630; Chamot, Pratt and Redfield, *J. Am. Chem. Soc.*, 1911, **33**, p. 366.

noldisulphonic acid (see Appendix B). Care should be taken in making this latter reagent as the results are dependent upon its composition.

Procedure. — For ground waters, measure with a pipette two samples of the water, one of two c.c. and the other of five c.c., into three-inch porcelain evaporating dishes and evaporate just to dryness on the steam bath or electric plate run at low heat. For surface-waters use 10 c.c. If the water is colored, decolorize with alumina as described under nitrites. Do not allow the residue to remain on the steam bath after all the water has been evaporated. Cool, add six drops of phenoldisulphonic acid and rub with a glass rod to insure complete contact of the acid and residue. Then add seven c.c. of distilled water and three c.c. of 30 per cent potassium hydroxide solution and mix thoroughly. A yellow color shows the presence of nitrates. Place this solution in a short Nessler tube* for comparison with a standard. This standard is prepared as follows: Place one c.c. of potassium hydroxide solution in a short Nessler tube and add standard nitrate solution from a burette until the color of the standard nearly matches that of the water sample. Make the volumes of the two solutions equal by diluting the standard and then add more standard nitrate solution until the colors exactly match. Use the sample of water for comparison which has the lighter color, unless there is no yellow at all. In case the five c.c. sample gives no color, repeat the determination, using 10 c.c. If this gives no color nitrates are absent. If the two c.c. sample gives a color which requires more than 10 c.c. of the standard, repeat the determination, using smaller amounts of water.

The standard nitrate solution contains 0.000001 gram N per c.c. From the amounts of standard nitrate solution and of water used calculate the amount of nitrate present expressed as nitrogen in parts per million of water.

Notes. — High chlorides seriously affect the accuracy of the method. This is noticeable in dealing with sea water and deep wells which contain large amounts of sodium chloride. In this

* An ordinary 50-c.c. Nessler tube cut off to a length of about five inches.

case, the reduction method with alkali and aluminum foil and distillation of the ammonia formed, is to be recommended.* For most drinking waters it is not necessary to use this method, which requires a much longer time than that described above.

Determination of Chlorine. — Chlorine is present in waters in the form of chlorides, and the term "chlorine" is used to mean "chlorides" as the results of analysis are given in terms of chlorine.

The determination is made by titration with silver nitrate in a solution alkaline with bicarbonates, — the condition generally existing in natural waters, — potassium chromate being used as an indicator.

Reagents. — The solutions required are a standard sodium chloride solution (1 c.c. contains 0.001 gram Cl), a solution of silver nitrate about one-half as strong, and potassium chromate indicator (see Appendix B).

Procedure. — Standardize the silver nitrate solution by titrating against a standard sodium chloride solution. To do this place 25 c.c. of distilled water in a 6-inch porcelain evaporating dish, add three drops of potassium chromate indicator, and then run in from a burette a measured amount of sodium chloride solution, about five c.c. being sufficient. It is not necessary to add exactly five c.c., but it is necessary to know the exact amount added. Now add silver nitrate solution from a burette until the yellow color of the solution has changed to a faint reddish brown. The end point is best seen if 25 c.c. of distilled water and three drops of indicator are placed in a 6-inch dish which is set beside the dish in which the titration is being carried on. This gives a standard color and the end point is reached when the solution being titrated shows the slightest appearance of red as compared with the standard. From the results of the standardization calculate the value of silver nitrate solution in terms of sodium chloride solution and in terms of Cl per c.c.

Test the water to be analyzed with phenolphthalein and with methyl orange. It should be acid to the former and alkaline

* See "Standard Methods," p. 25.

to the latter. If alkaline to phenolphthalein neutralize the sample measured for titration with dilute sulphuric acid. If acid to methyl orange neutralize with sodium bicarbonate.

Highly colored waters should be decolorized before titration as the color interferes with the end point. To do this shake some of the sample in an Erlenmeyer flask with milk of alumina, one c.c. of the latter being used for each 100 c.c. of water. Heat the mixture rapidly to boiling, allow to settle and decant through a filter.

Make a qualitative test for chlorides on the sample of water. If only a faint opalescence appears, a 250 c.c. sample must be used for analysis; if a marked cloudiness or a precipitate is formed, a 25 c.c. sample may be used. If the larger sample is found to be necessary, evaporate to about 25 c.c. on a steam bath or electric plate; avoid boiling. Cool before titrating.

To 25 c.c. of the water, measured with a pipette, or an evaporated 250 c.c. sample, in a 6-inch porcelain dish, add three drops of indicator and about five c.c. of sodium chloride solution, the exact amount being measured as in the standardization. Then run in silver nitrate solution until the end point is reached, using the standard color as before.

From the amounts of silver nitrate and sodium chloride solutions used calculate the amount of chlorine present in parts per million.

Notes. — With waters containing large amounts of chlorides, the addition of sodium chloride in the titration may be omitted.

It is important that the process be carried out essentially as described, since it has been found that the results vary with the volume of solution in which the titration is made, the amount of chromate used, and the amount of precipitated chloride present.*

Determination of the Carbonaceous Matter or "Oxygen Consumed." — This determination is supposed to give the amount of oxygen absorbed by the organic matter present in the water. Except in sewage analysis, the results are of little

* Hazen, *Am. Chem. J.*, 1889, 11, p. 409.

importance, and the determination may be omitted without appreciably affecting the interpretation of the results of the whole analysis.

The oxygen consumed is determined by allowing an excess of potassium permanganate in acid solution to act on the organic matter in the water under certain conditions, and then titrating the excess of permanganate with ammonium oxalate.

Equations:

$$4\ KMnO_4 + 6\ H_2SO_4 + 5\ C \rightarrow 2\ K_2SO_4 + 4\ MnSO_4 + 6\ H_2O + 5\ CO_2.$$

$$2\ KMnO_4 + 3\ H_2SO_4 + 5\ C_2H_2O_4 \cdot 2\ H_2O \rightarrow K_2SO_4 + 2\ MnSO_4 + 10\ CO_2 + 18\ H_2O.$$

Reagents. — The solutions required are a standard ammonium oxalate solution (1 c.c. equals 0.0001 gram oxygen), a potassium permanganate solution of approximately the same strength, and 1–3 sulphuric acid (see Appendix B).

Procedure. Kubel's Hot Acid Method. — Standardize the potassium permanganate against the oxalate in the following way: Measure 100 c.c. of distilled water into a 250-c.c. flat-bottomed flask, add 10 c.c. of sulphuric acid (1-3) and then add from a burette a measured quantity (about 10 c.c.) of standardized potassium permanganate solution. Place the flask on a wire gauze or electric stove and heat quickly to boiling. Boil the solution gently for exactly five minutes, remove it from the flame, cool for one minute, and add from a burette sufficient ammonium oxalate to decolorize the solution. Titrate back with the permanganate to a faint permanent pink color. Calculate the value of the permanganate in terms of standard ammonium oxalate and of oxygen.

For the analysis proceed just as in the standardization, replacing the distilled water by the sample to be tested. The oxygen consumed value for the water under examination is obtained from the number of c.c. of permanganate used in excess of that required to react with the oxalate added in the determination. Calculate the results in parts of oxygen per million of water.

Notes. — For highly colored surface-waters 25 c.c. are taken and diluted to 100 c.c. with water free from organic matter; for sewages, 10 c.c. or less are diluted in the same way.

The oxygen given up by the permanganate combines with the carbon of the organic matter and perhaps, to a certain extent, with the hydrogen, but not with the nitrogen. The amount of oxygen consumed bears some relation, therefore, to the amount of organic carbon present in the water, but this relation certainly cannot be taken as a definite one in every case, the results varying even with the time of boiling. The method has its greatest value when it is used to compare waters of the same general character and having the same origin; for example, in making periodical tests of the purity of the effluent from a filter. Furthermore, in order that the results shall have this comparative value, it is absolutely necessary that the process shall always be carried out in exactly the same way, even to the minutest detail of quantity, time and temperature.

In some cases it may be found advantageous to heat the solution upon the water-bath for half an hour instead of boiling it for five minutes. The results, however, will not be exactly comparable with those obtained by boiling.

Different kinds of organic matter behave differently with various oxidizing agents, so that a comparison of the results obtained with different oxidizing agents may throw light upon the character of the organic matter, as well as its amount.* In waters from the watersheds of eastern North America the color and the oxygen consumed have a certain, though somewhat varying, relation.

Determination of the Residue on Evaporation and the Loss on Ignition. — *Procedure.* — Carefully clean a large platinum dish, ignite for a few minutes over a burner, cool in a desiccator and weigh. Measure into it 100 c.c. of the water (200 c.c. in the case of surface-waters), and evaporate to dryness on the water-bath. When the water is all evaporated, heat the dish in the oven at the temperature of boiling water for one hour,

* Woodman, *J. Am. Chem. Soc.*, 1898, 20, p. 497.

cool in a desiccator over sulphuric acid, and weigh. The increase in weight gives the "total solids" or "residue on evaporation."

The residue should be ignited and the loss on ignition noted. Heat the dish in a "radiator," which consists of another platinum dish enough larger to allow an air-space of about half an inch between the two dishes, the inner dish being supported by a triangle of platinum wire. Over the inner dish is suspended a disc of platinum-foil to radiate back the heat into the dish. The larger platinum dish is heated to bright redness by a triple gas-burner. An electric muffle may be used in place of the radiator. This should be run at a temperature of about 500° C. Heat the dish until the residue is white or nearly so. Note any blackening or charring of the residue and any peculiar "burnt odor" which may be given off. After the dish has cooled, slightly moisten the residue with a few drops of distilled water. Heat the residue in the oven for an hour; cool in a desiccator and weigh. This gives the weight of "fixed solids," the difference being the "loss on ignition." Save the residue for the determination of iron.

Notes. — Before the introduction of modern methods of water-analysis, the determination of "loss on ignition" was the only method for the estimation of organic matter in water. In order, however, that the determination shall possess any real value, it is necessary to regulate carefully the heat during the ignition, so as to destroy the organic matter without decomposing calcium carbonate or volatilizing the alkali chlorides.

This is what the use of the radiator or muffle is intended to accomplish, and in the case of surface-waters with low mineral content and considerable organic matter, the method gives generally satisfactory results. But in the case of ground waters having little or no organic matter and high mineral content, the loss is often very great on account of the decomposition of nitrates and chlorides of the alkaline earths and the loss of water of crystallization. In waters of this class, the determination of "loss on ignition" is, therefore, generally meaningless, although an approximation to the amount of organic matter can be ob-

tained by the addition of sodium carbonate to the water before evaporating to dryness. By this means, the alkaline earths are precipitated as carbonates, the chlorine and nitric acid are held by an alkaline base, and there is no water of crystallization in the residue. Even with this modification, the loss is considerable when magnesium salts are present, owing to the evolution of carbonic acid.

It is the practice in some laboratories to ignite over a direct flame, taking care that the dish does not reach a temperature above a faint redness.

The behavior on ignition is oftentimes significant. Swampy or peaty waters give a brownish residue on evaporation to dryness, which blackens or chars, and this black substance burns off quite slowly. The odor of the charring is like that of charring wood or grain; sometimes sweetish, but not at all offensive. Waters much polluted by sewage blacken slightly; the black particles burn off quickly and the odor is disagreeable. Any observations on this point should be recorded in the report.

Determination of Iron. — This depends on the color produced by the action of potassium sulphocyanate on ferric chloride. The color obtained is compared with standards.

Reagents. — The solutions needed are a 1–1 hydrochloric acid, a potassium sulphocyanate solution, and a standard iron solution made from ferrous ammonium sulphate, one c.c. of this containing 0.0001 gram iron (see Appendix B).

Procedure. — Treat the residue from the loss on ignition, or that obtained by the evaporation of 100 c.c. of the water, with five c.c. of 1–1 hydrochloric acid, warming on the steam-bath or hot plate so as to dissolve as much as possible of the mineral matter. Wash the solution with distilled water into a 100-c.c. Nessler tube, filtering if there is any insoluble matter. Make up to about 50 c.c. with distilled water. Add potassium permanganate solution, a few drops at a time, until the solution remains pink for 10 minutes. This is to oxidize any ferrous chloride to the ferric condition. Then add 10 c.c. of potassium sulphocyanate solution and make the volume up to the 100 c.c.

mark with distilled water. Iron gives a red color. If iron is present prepare a blank standard by placing 75 c.c. of distilled water, five c.c. of hydrochloric acid and 10 c.c. of potassium sulphocyanate solution in a 100 c.c. Nessler tube. Now add from a burette, standard iron solution until the color nearly matches that obtained in the determination. Fill the tube with distilled water to the 100 c.c. mark and continue adding the iron solution until the color of the blank exactly matches that of the determination. From the number of c.c. of standard iron solution used calculate the amount of iron in the water.

Notes. — In the case of some river-waters, it will be found necessary to add a few cubic centimeters of hydrochloric acid to the water while evaporating, in order to facilitate the solution of the iron. This should be done on a separate portion from that used for the determination of total solids.

The colors should be matched immediately after adding the sulphocyanate, since the color fades appreciably on standing. If the color is greater than that given by 3.5 c.c. of the standard solution an aliquot part should be used. In this case sufficient hydrochloric acid and potassium sulphocyanate should be added so that the same amounts of these are present as given in the above directions.

Determination of Hardness. — *Soap (Clark's) Method.* This method really gives the soap consuming power and not the true total hardness, but it is in general use for sanitary purposes, and where the water is to be used for household purposes only, really gives what is most wanted. The determination depends on the fact that soap forms an insoluble precipitate with the calcium and magnesium salts in the water. As soon as the precipitation of the latter is complete a permanent lather is formed. This serves as the end point. The hardness is expressed in terms of calcium carbonate per million.

Reagent. — A standard soap solution (see Appendix B).

Procedure. — Measure 50 c.c. of water into a 200-c.c. clear glass-stoppered bottle and add the soap solution from the burette, two or three tenths of a cubic centimeter at a time, shaking well

after each addition, until a lather is obtained which covers the entire surface of the liquid with the bottle lying on its side, and is permanent for five minutes. The number of parts of calcium carbonate corresponding to the volume of soap solution used is found in the table in Appendix A.

Notes. — The importance of adding the soap in small quantities cannot be too strongly emphasized, especially in the presence of magnesium compounds. The presence of magnesium salts will be recognized by the peculiar curdy appearance of the precipitate formed and by the occurrence of a false end point, the lather lasting about three minutes when the titration is about half done.

By reference to the table it will be observed that values are not given for more than 16 c.c. of the soap solution. If in any case the water under examination requires more than 10 c.c. of the standard soap solution, a smaller portion of 25 c.c., 10 c.c. or even two c.c., as the case may require, is measured out and made up to a volume of 50 c.c. with recently distilled water. If the volume of soap used is always about seven c.c., this will keep the results comparable with each other, although the element of dilution introduces an error. Potable waters, in the eastern United States, at least, are rarely so high in mineral matter as to require excessive dilution. In the case of extremely hard waters, however, the acid method is to be preferred. Distilled water itself, containing no calcium salt whatever, requires the use of a considerable quantity of soap to produce a permanent lather. The cause for this seems to exist in the dissociation of the greater part of the soap at the extreme dilution to which it is subjected, and the slow accumulation of a sufficient quantity of undissociated soap to allow of the increase of surface tension to a point at which soap-bubbles will persist.

*Hehner's Acid Method.** — The temporary hardness of a water is that part of the total hardness which can be removed by boiling. It is due to the presence of the bicarbonates of calcium

* Hehner, *Analyst*, 1883, **8**, p. 77; Draper, *Chem. News*, 1885, **51**, p. 206; Ellms, *J. Am. Chem. Soc.*, 1899, **21**, p. 239.

and magnesium. These give an alkaline reaction to indicators such as methyl orange and erythrosine, and can be titrated with standard acid. The results obtained will differ slightly from the true temporary hardness, on account of the solubility of calcium and magnesium carbonates which are formed when a solution of the bicarbonates is boiled, but the results are close enough for practical purposes.

Permanent hardness is that which is not removed by boiling, and is due mainly to the presence of the sulphates and chlorides of calcium and magnesium. After removing the temporary hardness by boiling, the permanent hardness, i.e., the calcium and magnesium remaining in solution, may be determined by adding standard "soda reagent" (a mixture of equal parts of sodium hydroxide and sodium carbonate), which precipitates the magnesium as hydroxide and the calcium as carbonate. The excess of soda reagent added is then determined by titration with standard acid, — the amount consumed representing the calcium and magnesium.

If the original water is neutralized with sulphuric acid all the temporary hardness will be converted to permanent hardness. If this latter is then determined, it will represent the total hardness of the sample of water.

Reagents. — The solutions required for the hardness determinations are N/20 and N/50 sulphuric acid, N/10 soda reagent, methyl orange indicator and, for some purposes, erythrosine.

Procedure for Alkalinity. — Measure 200 c.c. of the sample, filtered if necessary, into a porcelain evaporating dish, add two drops of methyl orange indicator and titrate to a faint pink with N/50 sulphuric acid. The end point can best be seen by placing 200 c.c. of distilled water in another dish and adding two drops of indicator. This gives a standard color and the first change of the sample being titrated, toward a pink color, can be readily recognized. The number of c.c. of acid used multiplied by five gives the alkalinity in parts of calcium carbonate per million. Save the titrated sample for the determination of total hardness.

If the soap hardness is over 300, a 100 c.c. sample should be

used. In this case multiply the c.c. of acid by 10 to get the alkalinity.

Notes. — If the water to be tested has been treated with alum, erythrosine indicator must be used as methyl orange is not sufficiently sensitive. For this, measure 100 c.c. of the water into a clear bottle such as is used for the soap test, and add 2.5 c.c. of the erythrosine indicator (0.1 gram of the sodium salt in one liter of distilled water), and five c.c. of chloroform neutral to erythrosine. Mix well by shaking and add N/50 sulphuric acid from a burette in small quantities, shaking thoroughly after each addition. The pink color in the water gradually grows lighter until the addition of a drop or two of the acid causes it to disappear entirely. Make a correction for the indicator by carrying out a blank determination with distilled water. Multiply the c.c. of acid used by 10 to get the alkalinity in terms of calcium carbonate.

Procedure for Permanent Hardness. — Measure 200 c.c. of water into an Erlenmeyer flask, boil 10 minutes to expel carbon dioxide, and add 25 c.c. of N/10 soda reagent. For waters with a soap hardness over 300 use a 100 c.c. sample. Boil down to a volume of about 100 c.c., cool to 20° C., rinse into a 200 c.c. calibrated flask with cooled, boiled distilled water, and make up to 200 c.c. Mix thoroughly. Filter through a dry filter paper, receiving the filtrate in a 100 c.c. calibrated flask. Discard the first 30 or 40 c.c., and then collect 100 c.c. of the filtrate. Pour into an Erlenmeyer flask, add one drop of methyl orange indicator and titrate with N/20 sulphuric acid.

Make a blank determination with 200 c.c. of distilled water in place of the sample.

The difference between the amount of acid required by the blank and that required in the determination represents the amount of soda reagent used to precipitate the calcium and magnesium. To get the permanent hardness multiply this difference by 25 when a 200 c.c. sample of water is used.

If a water contains sodium or potassium carbonate, there will not be any permanent hardness, and hence more acid will be

required for the filtrate than corresponds to the amount of soda reagent added. From this excess the amount of sodium carbonate in the water may be determined. Any alkali carbonate present would be calculated as temporary hardness by the direct titration; hence it should be calculated to calcium carbonate and subtracted from the results found by the direct titration.

Procedure for Total Hardness. — Boil down the neutralized sample obtained at the end of the alkalinity determination to about 100 c.c., add 25 c.c. soda reagent, again boil down to 100 c.c., and proceed as in the determination of permanent hardness. The calculations are the same as described there.

Free Carbonic Acid. — This determination depends on the reaction of sodium carbonate with carbon dioxide to form the bicarbonate,

$$Na_2CO_3 + H_2O + CO_2 \rightarrow 2\,NaHCO_3.$$

As soon as all the free carbonic acid has been used up, the next drop of sodium carbonate will color phenolphthalein red.

Reagents. — These are an N/22 sodium carbonate solution, and phenolphthalein indicator.

Procedure. — Measure 100 c.c. of the sample into a tall, narrow vessel, preferably a 100 c.c. Nessler tube, add a few drops of phenolphthalein and titrate rapidly with N/22 sodium carbonate solution, stirring gently until a faint but permanent pink color is produced.

The number of c.c. of N/22 sodium carbonate solution used in titrating 100 c.c. of water, multiplied by 10, gives the parts per million of free carbonic acid as CO_2.

Note. — Owing to the ease with which free carbonic acid escapes from water, particularly when present in considerable quantities, it is highly desirable that a special sample should be collected for this determination, which should preferably be made on the ground. If this cannot be done, approximate results from water not high in free carbonic acid may be obtained from samples collected in bottles which are completely filled so as to leave no air space under the stopper.

Determination of Sulphates.* — Sulphates can be determined with an accuracy sufficient for most purposes by means of the Jackson Candle Turbidimeter.† The results are determined by the amount of turbidity produced by precipitated barium sulphate.

Procedure. — To about 100 c.c. of the water add sufficient dilute hydrochloric acid (about one c.c.) to acidify and then one-half a gram of barium chloride. Shake until dissolved. Pour slowly into the graduated tube of a candle turbidimeter until the image of the flame beneath just disappears. Read the height of the liquid in the turbidimeter tube and obtain from the table in Appendix A the parts per million of sulphates as SO_3.

Notes. — Care should be taken to have the solution well stirred before adding to the turbidimeter tube. The tube must not be placed over the flame when empty. Waters containing from 30 to 200 parts per million may be read directly; otherwise the water should either be concentrated or diluted.

Determination of Alum. — On account of the use of alum or aluminum sulphate as a coagulant in the filtration of water, a determination of alumina in the effluent water is often necessary. This may be readily made by the logwood test.‡

Procedure. — The logwood solution is made as follows: Take two grams of logwood chips and boil one minute in a platinum dish with 50 c.c. of distilled water. Decant the solution and boil again for one minute with 50 c.c. of water. Decant this and similarly boil a third time with 50 c.c. of water. Decant this into a platinum receptacle for use. Take three drops for each test. Kept in platinum, the solution will last for several days at least.

Test the water as follows: Boil 50 c.c. of the water in a platinum dish for a short time to expel carbon dioxide. Add three

* "Laboratory Notes on Industrial Water Analysis," Ellen H. Richards, 1910. J. I. D. Hinds, *J. Am. Chem. Soc.*, **18**, 661 and **22**, 269; D. D. Jackson, *J. Am. Chem. Soc.*, 1901, p. 799; Muer, *J. Ind. Eng. Chem.*, 1911, **3**, p. 553.

† For a description of this see references.

‡ E. H. Richards, *Tech. Quart.*, 1891, **4**, p. 194; A. H. Low, *Tech. Quart.*, 1902, **15**, p. 351.

drops of the logwood solution and continue boiling for a few seconds to develop the color. Decant into a glass flask and cool quickly under the tap (so as not to keep the hot solution too long in the glass). Transfer to a No. 2 beaker and blow in carbon dioxide from the breath by means of a glass tube until there is no further decolorization. Pour the water into a Nessler tube for comparison with standards similarly prepared from a standard alum solution. Allow them to stand several hours before taking the final reading. No wash-water is used at any of the decantations. The test shows one part of aluminum sulphate in 8,000,000 parts of water.

Notes. — A blank made with distilled water, if not completely decolorized by the CO_2, will show a tint perceptibly fainter than that produced by one part in 8,000,000 of aluminum sulphate.

It should be noted that carbon dioxide must be kept absent until the point prescribed. The solution is, therefore, transferred to a beaker in order to keep the flask free from carbon dioxide for the next test.

The main points are:

1. Any kind of logwood appears to answer.

2. The solution is good for several days, at least, if kept in platinum.

3. The use of platinum instead of glass for boiling the test solution.

4. The use of carbon dioxide instead of acetic acid.

Aluminum hydrate, as pointed out in 1893 by the late Professor A. R. Leeds, will produce a tint almost as strong as if it were in solution, but of a distinctly differing tint.

Low's method of procedure is as follows: First, test the water as above described. If no tint, or none exceeding that of the blank, remains after standing several hours or over night, that is sufficient. If, however, a tint persists, or a colored precipitate settles out, it is necessary to determine if this is due to aluminum hydrate. Pour a sample of the water several times through a double Swedish filter, and finally test the filtrate. If the tint produced is weaker than that given by the unfiltered

water, repeat the operation on a fresh portion of the water, using the same filter, and continue repeating with new portions of the water and always using the same filter, until it is apparent that no further diminution of the tint can be effected.

For a less delicate test in school laboratories where platinum is not available, the following alternative method may be used:

Dissolve about 0.1 gram pure hæmatoxylin in 25 c.c. water; this solution will keep for two weeks and works best after being made several hours. To 50 c.c. of the water, placed in a four-inch porcelain dish, add two drops of the hæmatoxylin solution, allow the solution to stand for one or two minutes, then add a drop of 20 per cent acetic acid. The standards are prepared at the same time, using 50 c.c. of distilled water and the required amount of a standard alum solution. The comparison must be made immediately, since the color fades on standing. In this way the presence of one part of aluminum sulphate in five million can be determined directly in the water and with ease.

Logwood may be used instead of the hæmatoxylin, the solution being prepared as above.

This test will show the presence of all soluble salts of aluminum which enter into combination with the coloring matter of the logwood to form a "lake."

The alkalies and alkaline earths give a purplish color with logwood extract, hence the test for alum can be made only in acid solution.

Determination of Lead. — Lead in the minute quantities in which it ordinarily occurs in water is best estimated by comparing the color of the sulphide with standards.

Procedure. — If the water is colorless, fill a 100 c.c. Nessler tube to the mark, acidify with a few drops of acetic acid, and add from a glass tube one drop of calcium sulphide solution. A black tint to the precipitated sulphur shows the presence of lead. A quantitative estimate may be made by comparison with a series of standards made from a standard lead solution.

If the water is too highly colored to estimate the lead directly, evaporate three or four liters in a porcelain dish to about 25 c.c.,

add 10 c.c. of ammonium chloride solution and a considerable excess of strong ammonia. Then add hydrogen sulphide water and allow the dish to stand some hours. Boil the contents of the dish for a few moments to expel the excess of hydrogen sulphide, and filter. The precipitate contains all the lead, iron, and suspended organic matter, also copper and zinc if present, while the soluble color goes into the filtrate. Wash once with hot water, transfer the filter to the original dish, and dissolve the sulphides by boiling with dilute nitric acid (1 part acid, sp. gr. 1.2, to 5 parts water). Filter and wash; evaporate to 10–15 c.c., cool, add 5 c.c. concentrated sulphuric acid and evaporate until copious fumes are given off. Then, if the original water contained less than 0.25 part iron per million, add acetic acid and ammonia, boil, filter and read the amount of lead in the alkaline filtrate, making the standards also alkaline with ammonia.

If the water contained over 0.25 part iron, wash the lead sulphate into a beaker with alcohol and water, and let it settle overnight. Filter, wash free from iron with 50 per cent alcohol, dissolve the precipitate by boiling with ammonium acetate, filter, and determine the lead as above.

Note. — If more than 0.25 part of iron is present, some of the lead will be held by the precipitated ferric hydroxide; and if 25 parts are present, all of the lead may be lost in this way; hence the modification of the method in the presence of considerable quantities of iron.*

When copper is also present it is detected by the blue color given to the ammoniacal filtrate from the iron precipitation.

Determination of Phosphates.† — *Procedure.* — Evaporate 50 c.c. of the water and three c.c. of nitric acid (sp. gr. 1.07) to dryness in a three-inch porcelain dish on the water-bath. Heat the residue in an oven for two hours at the temperature of boiling water. Treat the dry residue with 50 c.c. of cold distilled water,

* Ellms, *J. Am. Chem. Soc.*, 1899, **21**, p. 359.

† Lepierre, *Bull. Soc. Chim.*, 1896, **15**, p. 1213; Woodman and Cayvan, *J. Am. Chem. Soc.*, 1901, **23**, p. 96; Woodman, *ibid.*, 1902, **24**, p. 735.

added in several portions and poured into the comparison-tube. It is not necessary to filter the solution. Add four c.c. of ammonium molybdate (50 grams per liter) and two c.c. of nitric acid, mix the contents of the tube and compare the color, after three minutes, with standards made by diluting varying quantities of the standard phosphate solution (1 c.c. = 0.0001 gram P_2O_5) to 50 c.c. with distilled water and adding the reagents as above. Carry out a blank determination on the distilled water used for dilution, especially if it has stood for any length of time in glass vessels.

Notes. — The method as described will be sufficient for ordinary work. If a more exact determination of the phosphate is required, a slight correction should be made in each case. For a table showing these corrections reference may be made to the paper by Woodman and Cayvan previously cited.

The evaporation and heating with nitric acid is for the purpose of removing silica, which gives with ammonium molybdate a yellow color similar to that given by phosphates.

The determination of phosphates in a drinking-water is a matter which has not received the attention from water analysts that has been given to the estimation of various other constituents. Any one who looks through the literature cannot help noticing how few are the published results of quantitative estimations of the phosphate content of natural waters, apart from mineral waters. Yet this determination, by reason of the conversion of organic phosphorus compounds into phosphates through the process of decay, is one which might reasonably be expected to throw considerable light on the question of the pollution of natural waters by objectionable material.

The reasons for this dearth of published data are not far to seek. To be of value the amount of phosphate must be known within rather narrow limits. Qualitative tests are not sufficient. The mere presence of phosphates is by no means definite or even confirmatory evidence of organic pollution. Rocks and minerals containing phosphates are found nearly everywhere, and traces, at times even considerable quantities, may be dis-

solved, especially by waters rich in carbonic acid. This, however, does not constitute a serious objection to the utility of the determination. The same is true of many, if not most, of the constituents upon which reliance is placed in judging of the quality of a water. Unpolluted waters often contain notable amounts of nitrates and chlorides, and a true judgment can be rendered only after comparison with samples from adjacent but unpolluted sources.

The chief reason, however, has been the lack of an accurate and simple method, sufficiently delicate, and of enough data to work out a standard for comparison.

This reason can hardly hold true now, for enough work has been done on the colorimetric method to indicate its value as another link (of which we have none too many, anyway) in the chain of circumstantial evidence by which we are often compelled to judge the purity of a water.

The amount of phosphate and its variation seem to follow the same general line as the other mineral constituents which either accompany the polluting material or are produced by its decay, especially the nitrates and chlorides. It is not, however, so delicate an indicator as these. In general, it may be said that the amount (expressed as P_2O_5) in an unpolluted water will seldom be over 1.0 part per million.

Determination of Dissolved Oxygen. — *Winkler Method.** — The method depends on the absorption of oxygen by manganous hydroxide with the formation of manganese dioxide; the liberation of iodine by this last in an acid solution containing potassium iodide; and the titration of the iodine with sodium thiosulphate. The reactions involved can be expressed as follows:

$$MnSO_4 + 2\,NaOH \rightarrow Mn(OH)_2 + Na_2SO_4.$$

$$2\,Mn(OH)_2 + O_2 \rightarrow 2\,MnO_2 + 2\,H_2O.$$

$$MnO_2 + 2\,H_2SO_4 + 2\,KI \rightarrow MnSO_4 + I_2 + K_2SO_4 + 2\,H_2O.$$

$$2\,Na_2S_2O_3 + I_2 \rightarrow 2\,NaI + Na_2S_4O_6.$$

* Berichte, 1888, **21**, p. 2843; also see "Standard Methods of Water Analysis."

The method has recently been modified by Hale and Melia* by titrating the iodine in an acetic acid solution in order to avoid difficulties due to the presence of nitrites and nitrates, and this should be followed in testing for putrescibility.

Collection of Samples. — The samples are collected in glass-stoppered bottles of known capacity, holding about 300 cubic centimeters. When water is taken from a faucet, the bottle is filled by means of a tube which passes to the bottom of the bottle. A considerable amount of water is allowed to pass through the bottle and overflow at the top. It will be almost impossible to obtain duplicate samples unless the bottles are filled at the same time by means of a T tube, owing to variations in pressure in the pipes.

In taking samples from a stream or pond, a stopper with two holes is used. A tube passing through one of these holes is sunk in the water to the desired depth, and the other is connected with a larger bottle of at least four times the capacity of the smaller one, and fitted in the same way. From the larger bottle the air is exhausted by the lungs or by an air-pump until it is nearly filled with water. Unless the determination is to be made at once, the rubber stopper of the smaller bottle is quickly replaced by the glass stopper so that no air is left in the bottle. The temperature of the water at the time of sampling should be noted.

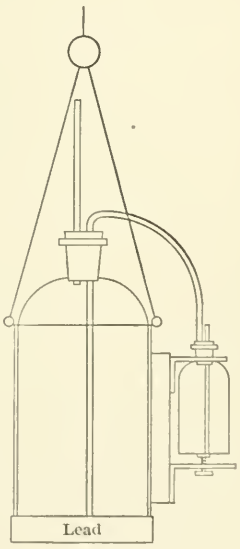


FIG. 10.

The apparatus which has been used in connection with work in this laboratory for collecting samples at various depths down to 75 feet is shown in outline in Fig. 10. A galvanized-iron can of such size as to hold one of the gallon bottles is weighted with lead and provided with ears at the top for suspending. The

* *J. Ind. Eng. Chem.*, 1913, 5, p. 976.

bottle, which is securely wired in, is provided with a rubber stopper carrying two brass tubes, one ending just below the stopper and projecting for about 8 or 9 inches above it, the other extending to the bottom of the bottle and connected by heavy rubber tubing with the sample bottle. This is held by brass brackets, which are fastened by means of a wooden cleat to the side of the can. The neck of the bottle is put into the slot in the upper bracket and then it is firmly clamped by the thumb-screw of the lower one. The arrangement of tubes in the sample bottle is obvious. In using the apparatus it is quickly lowered to the desired depth by means of a rope marked off in feet. The water enters the sample bottle and flows through it into the other. When the bubbles cease to rise, indicating that the larger bottle is full, thus replacing the water in the sample bottle a number of times, the apparatus is drawn to the surface. The temperature is read from a thermometer fastened to the tube inside the gallon bottle.

Reagents. — The reagents needed are solutions of manganous sulphate, potassium iodide in sodium hydroxide, potassium acetate, N/100 sodium thiosulphate, and starch indicator (see Appendix B).

Procedure. — Remove the stopper from the 300-c.c. calibrated bottle, and add two c.c. of manganous sulphate solution with a pipette having a long capillary point reaching to the bottom of the bottle, and in the same way add two c.c. of the solution of sodium hydroxide and potassium iodide. Insert the glass stopper, leaving no bubbles of air, and mix the contents of the bottle. Allow the precipitate to settle, remove the stopper and add two c.c. of concentrated hydrochloric acid from a pipette in the same manner as before. Replace the stopper, driving out some of the liquid, and shake until the precipitate is dissolved, and the liquid homogeneous. Remove 100 c.c. with a pipette or graduated flask, and titrate with N/100 sodium thiosulphate, using starch as an indicator. Add the starch solution, about two c.c., only after the iodine solution has become a light straw color.

To calculate the results proceed as follows: Let V equal the volume of the bottle with the stopper inserted and N the number of c.c. of thiosulphate used. One c.c. of N/100 sodium thiosulphate is equivalent to 0.00008 gram of oxygen. The actual volume of water from which the oxygen was removed is equal to the volume of the bottle minus the four c.c. displaced by the first two reagents added. The liquid displaced by the acid does not need to be allowed for, as it did not contain any oxygen or iodine. The oxygen equivalent to the iodine titrated in the 100 c.c. of the solution removed is equal to $N \times 0.00008$.

The oxygen equivalent to the total iodine liberated is equal to

$$\frac{N \times 0.00008 \times V}{100}.$$

This is the oxygen present in the original water, which has a volume of $(V - 4)$, the four c.c. being the part displaced by the solutions added. The oxygen in parts per million is, therefore, equal to

$$\frac{N \times 0.00008 \times V \times 1{,}000{,}000}{100 \times (V - 4)} = \frac{0.8\,NV}{V - 4}.$$

If the sodium thiosulphate solution is not exactly N/100, the correct oxygen equivalent should be substituted in place of the value 0.00008.

QUANTITIES OF DISSOLVED OXYGEN IN PARTS PER MILLION BY WEIGHT IN WATER SATURATED WITH AIR AT THE TEMPERATURE GIVEN

Temp. C.	Oxygen.	Temp. C.	Oxygen.	Temp. C.	Oxygen.	Temp. C.	Oxygen.
0	14.70	8	11.86	16	9.94	24	8.51
1	14.28	9	11.58	17	9.75	25	8.35
2	13.88	10	11.31	18	9.56	26	8.19
3	13.50	11	11.05	19	9.37	27	8.03
4	13.14	12	10.80	20	9.19	28	7.88
5	12.80	13	10.57	21	9.01	29	7.74
6	12.47	14	10.35	22	8.84	30	7.60
7	12.16	15	10.14	23	8.67	.....	.....

The results of this determination are frequently expressed in per cent of saturation, which is given by the ratio of the oxygen found to that present if the water were completely saturated at the same temperature. The latter figure is given by the preceding table.

*Procedure to be Followed in Putrescibility Tests or with Polluted Waters.** — Follow the directions as given above until after the addition of the concentrated hydrochloric acid. Then replace the stopper and shake until all the precipitate is dissolved. Remove the stopper and add from a pipette two c.c. of potassium acetate solution. Mix by pouring into a flask or beaker and back into the bottle. Remove 100 c.c. as before and titrate with N/100 sodium thiosulphate.

The addition of the acetate increases the volume of the iodine solution from V (the volume of the bottle) to $(V + 2)$, and this should be substituted in the formula given on p. 103. The oxygen in parts per million will, therefore, be equal to

$$\frac{0.8\,N\,(V+2)}{V-4}.$$

Notes. — If water is collected in the ordinary way and transferred to the apparatus by pouring, there will inevitably be an absorption of oxygen unless the water is already saturated. Thus a process which gives excellent results when the water is nearly or quite saturated may fail entirely to give accurate results when the dissolved oxygen is low or absent. The water may be supersaturated with oxygen in which case the per cent of saturation may be more than 100.†

Determinations of dissolved oxygen in ponds and streams are best made on the spot, or, at least, the reagents should be added until after the addition of the hydrochloric acid. The very simple apparatus required for the Winkler process can be packed in a small space, and the entire determination requires only a few minutes. The absorption of the oxygen by the manganous

* See article by Hale and Melia, *loc. cit.*

† Gill, *Tech. Quart.*, 1892, **5**, p. 250.

hydroxide is complete almost at once, and it is unnecessary to allow it to settle for a long time before adding the acid. The titration can be made with a small burette or pipette with accurate results.

Putrescibility Test.* — There is at the present time no really satisfactory standard putrescibility test. One which seems to have been worked out on logical principles and which has given satisfaction in this laboratory is that proposed by the Royal Sewage Commission. The putrescibility is measured by the absorption of dissolved oxygen under given conditions. A stream water or diluted sewage or effluent, — the water used for dilution furnishing the necessary oxygen, — is tested for dissolved oxygen. A sample is then incubated in a closed bottle for five days at 20° C. and the dissolved oxygen again determined. The difference represents the oxygen absorbed, and should not be greater than 20 parts per million.

Procedure. — If the water is from a stream or lake, fill completely two 300-c.c. calibrated glass-stoppered bottles. Insert the stoppers, taking care that no air bubbles are enclosed. If a sewage or effluent is being tested, make dilutions with tap water as follows:

Raw sewage. Dilute 6 c.c. to 600 c.c.
Settling tank effluents. Dilute 12 c.c. to 600 c.c.
Filter effluents. Dilute 120 c.c. to 600 c.c.
Fill two calibrated bottles as just described.

Make a dissolved oxygen test on one bottle, following the directions as given for putrescibility tests. Set the other bottle in a 20° incubator and let stand for five days. Then determine the dissolved oxygen again. Calculate the results in terms of oxygen absorbed by the original sample of water, sewage or effluent.

Determination of the Color. — The amount of color is generally determined by direct comparison of the water with some definite standard of color. Various standards have been pro-

* *Eng. Rec.*, 1913, 68, pp. 315 and 453; *Am. J. Pub. Health*, 1914, 4, p. 241.

posed, the objection to most of them being that they are not sufficiently general in their application, being adapted only for the color of some particular class of waters.

The standard in most general use is the platinum standard. The comparisons of the water with the color standards are most readily made in 50-c.c. Nessler tubes. According to this scale, the color of a water is the amount of platinum in parts per million, which, together with enough cobalt to match the tint, must be dissolved in distilled water to produce an equal color. In practice, a standard having a color of 500 is prepared by dissolving 1.246 grams of potassium platinic chloride (equivalent to 0.5 gram platinum), 1.0 gram of cobalt chloride (equivalent to 0.25 gram cobalt), and 100 c.c. of strong hydrochloric acid in distilled water and diluting to one liter.

Dilute standards for use are made by diluting varying amounts of this standard to 50 c.c. with distilled water. Thus, by diluting one c.c., two c.c., and three c.c. each to 50 c.c., colors of 10, 20, and 30 are obtained. It is claimed that the platinum standards are permanent if protected from the dust, but in this laboratory it has been found necessary to replace them about once a month.

Determination of the Odor. — *Cold.* — Shake violently the sample in one of the large collecting-bottles when it is about half or two-thirds full, then remove the stopper and quickly put the nose to the mouth of the bottle. Note the character and degree of intensity of the odor, if any. An odor can be often detected in this way which would be entirely inappreciable if the water were poured into a tumbler.

Hot. — Pour into a plain beaker about five inches high enough water to one-third fill it. Cover the beaker with a well-fitting watch-glass and place it on an iron plate which has been previously heated, so that the water shall quickly come to a boil. When the air bubbles have all been driven off and the water is about to boil, take the beaker from the plate and allow it to cool for about five minutes. Then shake it with a rotary movement, slip the watch-glass to one side and put the nose into the beaker.

Note the odor as before. The odor may or may not be the same as that of the water when cold; it can be perceived, as a rule, for only an instant.

Notes. — It is inevitable that a certain personal equation should influence this test. Each laboratory will have its own standards for routine work, but a certain familiarity with the more common odors will tend to allay public anxiety and to aid in a more watchful habit on the part of consumers. Good ground waters do not give distinct odors unless they are derived from clayey soil, but the odor often betrays a contaminated well. Surface-waters will nearly always yield a characteristic odor. This odor may be due to the organic matter contained in the water, or to the presence of minute plants or animal organisms.

Among the odors which are frequently met are "earthy," "vegetable," "musty," "mouldy," "disagreeable," and "offensive." The "earthy" odor is that of freshly turned clayey soil. "Vegetable" is the odor of many normal colored surface-waters; it may be described as swampy or marshy, pond-like, and is often strengthened by heating. "Musty" can be likened to the odor of damp straw from stables; it is fairly characteristic of sewage contamination, and by the trained observer is distinctly distinguishable from the mouldy odor. "Mouldy" is the odor of upturned garden or forest mould, or of a moist hothouse; it is somewhat allied to the earthy odor. "Disagreeable" is a term which is capable of wide variation among different observers. It may include certain characteristic odors which are peculiar to the growth or decay of certain organisms, as the "pigpen" odor of *Anabæna*, the "fishy" or "cucumber" odor of *Synura*, etc. The term "offensive" is generally reserved for the sewages. These terms can be taken only as broad illustrations of the character of the particular odor, since the odor will very likely be described by different persons in different ways, and each laboratory will have its own characterization. The odor which often accompanies an abundant development of diatoms is a good illustration of this. It will be called by various

inexperienced observers offensive, rotten, fishy, geranium-like, aromatic, in one and the same sample of water.

The terms generally used to signify the degree of intensity of the odor are "very faint," "faint," "distinct," and "decided." The exact value to be placed upon each of these terms will, as a matter of course, vary with the individual analyst, but in a general way, it may be said that the "very faint" odor is one that would not be detected except by the trained observer; the "faint" odor would be recognized by the ordinary consumer if his attention were called to it; the "distinct" odor is one that would be readily noticed by the average consumer, but would not interfere with the use of the water; while the "decided" odor is one which would, in all probability, render the use of the water unpleasant.

Determination of the Turbidity and Sediment. — The suspended matter remaining in the water after it has rested quietly in the collecting-bottle for twelve hours, or more, is called its turbidity, and that which has settled to the bottom of the bottle, its sediment.

Good ground waters are often entirely free from turbidity and sediment, the suspended matters having been filtered out during the subterranean passage of the water, but this is rarely true of surface-waters. The turbidity is various in character and amount, sometimes milky from clay or ferrous iron in solution; usually it consists of fine particles, generally living algæ or infusoria. These often collect on the side toward or from the light, and a practiced eye can, not infrequently, recognize their forms. Some of the lower animal forms can also be seen by the naked eye, and the larger Entomostraca are quite noticeable in many waters.

The sediment may be earthy or flocculent; in the latter case it is generally débris of organic matter of various kinds. The degree of turbidity is expressed by the terms "very slight," "slight," "distinct," and "decided," and the degree of sediment by "very slight," "slight," "considerable," and "heavy." These determinations, again, are of value only to the routine

worker, and for him there are various methods in use. The papers of Parmelee and Ellms * and of Whipple and Jackson † should be consulted for a description of these.

Sewage Analysis.‡ — The methods for the analysis of sewages and sewage effluents are the same as those described for water. The main difference is in the quantities used for the various determinations. In most cases, this has been noted in connection with the analysis. Great care should also be exercised in taking samples from a bottle as the large amount of suspended matter makes it more difficult to obtain a representative portion. Special attention is called to the putrescibility test (p. 105) for effluents, as stability is the main desire in treating a large proportion of sewages.

Biological Examination. — Since a large number of, if not all, diseases are caused by living organisms, it would seem most desirable in examining a water supply if the specific organisms causing water-borne diseases could be looked for, and, if present, isolated. However, it is quite impossible to do this in the great majority of cases, and so in bacteriological work, just as in chemical analysis, certain indications of the presence of sewage are sought for, and if these indications are positive, the water is condemned. In a bacteriological examination, the most important index of the presence of sewage is finding *B. coli* in quantities as great as one in each cubic centimeter. This organism is a normal inhabitant of the intestines of man and the higher animals and is present in large numbers in human and animal excreta. Its presence, therefore, in water shows the presence also of sewage. For a discussion of water bacteriology and methods of analysis the reader is referred to another book.§

The close relation of the odor to the living flora and fauna of

* *Tech. Quart.*, **12**, 1899, p. 145.

† *Ibid.*, p. 283.

‡ See Fowler "Sewage Works Analyses," John Wiley & Sons, 1902.

§ Prescott and Winslow, "Elements of Water Bacteriology," 3rd edition, John Wiley & Sons, 1913.

the water makes it desirable that the chemist shall be able to recognize the more common forms of water plants and animals, even if he make no pretensions to a knowledge of cryptogamic botany or of zoology. Therefore, a microscope and a concentration apparatus should be in every water-laboratory. A full description will be found elsewhere.*

* Whipple, "The Microscopy of Drinking Water," 3rd edition, John Wiley & Sons, 1914.

CHAPTER VII

FOOD IN RELATION TO HUMAN LIFE: COMPOSITION, SOURCES, DIETARIES

LIFE itself is conditioned on the food-supply. Wholesome food is a necessity for productive life. Man can and does exist on very unsuitable, even more or less poisonous, food, but it is merely *existence* and not effective life. This is true not only of the wage-earner, but of the business-man, the professional man, the scholar. To be well, to be able to do a day's work, is man's birthright. Nevertheless, a too large proportion of the American people sells this most valuable possession for a mess of pottage which pleases the palate for three minutes and weights the digestive organs for three hours. With the products of the world exposed in our markets, the restraints of a restricted choice, as well as inherited instincts or traditions, lose their force. The buyer, unless he has actual knowledge to guide him, is swayed by the caprices of the moment or the condition of his purse, and often fails to secure adequate return in nutritive value for the money paid. The fact that so much *manipulated* material is put upon the market renders this choice of food doubly difficult, since the appearance of the original article is often entirely lost, and to city-bred buyers even the natural product conveys little idea of its money value. It is now even more necessary that an elementary knowledge of the proximate composition and food value of the more common edible substances should be recognized as an essential part of education.

Food: Definition and Uses. — Food is that which builds up the body and furnishes energy for its activities: that which brings within reach of the living cells which form the tissues the elements which they need for life and growth. Only such available substances can be called food, no matter what their chemical compo-

sition may be. Soft coal contains carbon and hydrogen and is food for the furnace, but is not available for the animal body.

This food which is taken into the body is used in various ways. It forms and builds up new tissues, besides repairing and making good the waste of tissues due to bodily activity; it is stored up in the body to meet a future demand; it supplies the needed heat by the transformation of its stored up or potential energy into the muscular energy required by the body; it may be used to protect the tissues of the body from being themselves consumed as food.

Composition of Food. — We determine what chemical elements enter into the composition of the body by an analysis of the various organs and tissues. We learn what combinations of these elements serve as food by determining those present in mother's milk and in foodstuffs which experience has proved to furnish perfect nutrition. From these studies it is apparent that about fifteen chemical elements are constant constituents of the human body; that about a thousand natural products are known to have food value; that of these, one hundred are of world-wide importance (see table, page 118), and that ten of them form nine-tenths of the food of the world.

The composition of food, as shown by chemical analysis, is not, however, the only factor that must be known to determine its value. The digestibility of the material must be taken into account as well. "We live not upon what we eat, but upon what we digest." It is more important to know the amount of *available nutrients* than the amount of total nutrients.

Food Principles. — While the foodstuffs present great variety, the food principles may be grouped under four headings; viz., nitrogenous substances or proteids, fats, carbohydrates, and mineral salts. Each group contains many members with minor but often essential differences. To make these substances available, there is needed an ample supply of air and of water, — of water for solution and circulation, of air for the oxygen needed to liberate the stored energy of the food in the place where it will accomplish its purpose.

Nitrogenous Substances. — Since, in some way as yet unknown to us, nitrogen is essential to living matter, such substances as contain this element in an available form are of the first importance. Some, as albumen, are so closely allied to human protoplasm that probably they need only to be dissolved to be at once assimilated. Others, as gluten and similar vegetable products, undergo a greater change; while still others, as gelatine, have a less profound but marked effect in protecting the tissues from waste.

The enzymes, "ferments," in part, of the older nomenclature, are also highly nitrogenous substances present in some form in nearly all foodstuffs of natural origin. The nearer the composition of the food approaches that of the protoplasmic proteid, presumably the greater its food value, since each cleavage, each hydrolysis, each step in the breaking down of the highly complex molecule, consisting of hundreds of atoms, is supposed to liberate the stored energy. Therefore, it is not a matter of indifference in what form this essential is taken. So little is known, however, with scientific accuracy that students will find a fruitful field of research along these lines of investigation. Also together with this element, nitrogen, go others, in small quantity to be sure, but evidently of great value. Such are sulphur, iron, phosphorus. One difference between the several groups of proteids is seen in this combination with the metallic elements which seems to carry with it certain effects. Until greater progress has been made in determining the availability in the organism of the various known substances, we must be content with a wide margin in the calculated quantities necessary for the daily efficiency, except in the very few instances of nearly pure substances, as white of egg. It is evident, also, that the manner of preparation and the kind of mixtures used in food will affect most profoundly so unstable and complex a class of substances. One thing is certain, that the body cannot take nitrogen from that which does not contain it. Therefore, a certain quantity of highly nitrogenous food should form a portion of the daily supply. It is usually held

that the body seems to be sufficiently nourished when the food contains an amount of digestible proteid equivalent to about 100 grams of dry albumen per day for the average adult, although recent work has shown that this figure is probably too high. An excess appears to have a stimulating effect and overloads the system with the waste, since the end-products are not purely mineralized substances, as are carbon dioxide and water from the carbohydrates, but are compounds of an organic nature, as creatin, urea, and uric acid, which have deleterious effects when accumulated in the system. A deficiency of nitrogen is made good, to a limited extent, by the protective agency of the other foodstuffs which offer themselves for all the offices except the final one of tissue-building.

Fats. — For this protective action, as well as for many other purposes, the fats are most valuable, and if they occur in about the same proportion as do the nitrogenous elements, the needs of the organism seem to be well met. Thus, in mother's milk, in eggs, and in meat from active animals these two are in nearly equal proportions, while in the cereals the fat is less; in nuts and in meat from fattened animals, as a rule, it is higher than the nitrogen. Little is known as to the varying food value of these fats from different sources. Certain physical conditions of solidity, melting-point, etc., seem to have more influence than mere chemical composition. Whatever the source, it is certain that the stored-up energy which is to serve the organism in cases of loss of income from any cause is in the form of fat, a form which is not subject to the action of agents which so readily decompose proteids and carbohydrates and yet is readily converted into available food whenever called for. That it is not absolutely necessary that the food should contain fat as such seems to be proved by experiment, but from the fact that nearly all natural food-substances do contain it, and that it appears to be more economical of human energy to take it from these foods than to manufacture it from the proteids and carbohydrates, we may safely assume fat to be an essential of the human dietary.

That the equality in amount of fat with nitrogenous compounds is not essential is proved by the fact that the strong draft animals, as horses and oxen, take food in which the per cent of fat is not more than half as much as of proteid; nevertheless, it is present in the food of all animals and doubtless, in its turn, is protected by an excess of the third class of foodstuffs, the carbohydrates, characteristic of the vegetable kingdom — a class which in the final decomposition, yields clean volatile products, water and carbon dioxide, and which, therefore, do not clog the system so readily as do urea and other wastes.

Carbohydrates. — The number of more or less well-defined substances under this head is legion: starches from scores of plants, sugars from as many more, gums, pectins, and dextrins, all with a certain food value, dependent probably upon the utilization of the various mixtures with which they are taken into the alimentary canal. These foodstuffs are very liable to "fermentation," that is, to an acid decomposition which prevents their absorption by the delicate lining of the walls of the intestines and which causes digestive disturbance. The sugars, which are very soluble, and, therefore, liable to be present in excess, are especially subject to this change. This class of food-substances is found in the diet of civilized man, free to choose, in an amount about equal to the sum of the other two classes, with a tendency to less rather than more. It may be said that sugar and fat increase over starch in the diet of a people of unrestricted choice, but it is not certain that the qualities of body which make for hardihood and resistance to disease are correspondingly increased. There is, indeed, much evidence to show that power of digesting vegetable foods indicates a general well-being of body conducive to long life. A ready adaptation renders possible the changes of habitat required by civilization. Unless one is to be confined to a narrow range, it is wise to cultivate a strength of digestion as well as a strength of muscle, and for the best brain power we believe it to be more essential.

Mineral Salts. — The fourth class, mineral salts, comes into the food largely from the vegetable substances eaten, for in these the union is an organic one readily assimilated. As we have seen, certain elements go with the nitrogenous portion, as, for example, in gluten and its congeners are found sulphur and phosphorus. Potassium, found in barley, is a constant constituent of protoplasm, while sodium is found in blood-serum. A lack of vegetable foods seems to impoverish the blood-corpuscles. For children, a deficiency in lime causes serious disease. Sugar, olive-oil, corn-starch and other prepared food-substances cannot take the place of asparagus, cabbage, carrots, etc.

To sum up briefly, then, we may say that the *protein* or nitrogenous portion of the food forms tissue, such as muscle, sinew and fat, and furnishes energy in the form of heat and muscular strength; the *fats* build up fatty tissue, but not muscle, and supply heat; the *carbohydrates* are changed into fat and supply heat. Another important use of the nutrients is to protect each other from being used in the body. The carbohydrates, especially, in this way protect the protein, including muscle, etc., from consumption.

Change in Composition Due to Cooking. — The composition of cooked foods is in general not the same as the raw material on account principally of chemical and physical changes brought about by the heat employed in the cooking process. The total nutrients, calculated on a water-free basis, may be practically the same, but the structure is often quite different.

Starch is hydrolyzed and rendered soluble by heating in the presence of moisture, and at higher temperatures it may be converted into the brown, soluble dextrin. The sugars are changed, being, in the case of sucrose, partly converted into other forms, such as invert sugar, by the heating, with the help of the organic acids present in many foods. Some of the proteids tend to become less soluble through heating and at higher temperatures may be even partly decomposed with possible loss of food value.

Heat of Combustion. — Until a more definite knowledge of the processes of metabolism (the transformations of matter and energy in the animal organism) is obtained the potential energy of food is calculated in terms of mechanical work — expressed in heat-units or calories.

One calorie is the amount of heat required to raise the temperature of one gram of water one degree centigrade. A gram of fat, as actually digested and oxidized in the body, affords enough heat to raise the temperature of about 9000 grams of water one degree. In like manner a gram of protein has an energy-producing power expressed in calories of about 4000, and for carbohydrates the average value is also 4000.

Allowance is made in these figures for the fact that to digest completely any part of our food results in a decrease of the amount of energy to be derived from it, and this affects the protein more than it does the other two. It is probably true that under favorable conditions the fat and carbohydrates can be completely utilized in the body and consequently their energy-producing power can be correctly estimated from their heat-producing power outside the body. In the case of protein, however, the digestion within the body is never so complete as to furnish all the energy that would be obtained by a complete combustion of these nitrogenous materials outside of the body.

The fact remains, however, that all experiments yet made go to show that within practical limits we are safe in using the heat of combustion (expressed in calories) of any food-substance as a controlling measure of food values.

Nutritive Ratio. — The requisite number of calories must, however, be obtained by the utilization of such substances as contain all the elements needed by the body, and in such ratio as has been found available for the balance of nutrition. In carrying on its multifarious activities the body loses about 20 grams of nitrogen per day, which must be replaced by the same element in the food taken. Thus while the requisite number of calories may be furnished by fat or starch, these substances alone will not suffice for

COMPOSITION OF SOME COMMON FOOD-MATERIALS AS PURCHASED

I. Fuel Value 3000–4000 Calories * per Pound

Food-material.	Refuse.	Water.	Nitrogenous Substances	Fat.	Carbohydrates.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Butter	.....	11.0	1.0	85.0	.....
Lard (refined)	.....	.....	.....	100.00	.....
Oleomargarine	.....	9.5	1.2	83.0	.....
Salt fat pork	.....	0.3 to 12.2	0.2 to 5.0	80.3 to 94.1	.....
Suet	.....	4.3 to 21.9	1.1 to 7.5	70.7 to 94.5	.....
Walnuts (shelled)	.....	2.5	16.6	63.4	16.1

II. Fuel Value 2000–3000 Calories per Pound

Bacon	8.7	18.4	9.5	59.4	.....
Cheese (American pale)	.....	31.6	28.8	35.9	0.3
Chocolate	.....	1.5 to 10.3	12.5 to 13.4	47.1 to 50.2	26.8 to 33.8
Doughnuts	.....	11.0 to 25.8	5.1 to 7.6	16.4 to 25.7	45.8 to 63.2
Mutton flank (fat)	.....	28.9	10.7	59.8	.....
Peanut butter	.....	2.1	29.3	46.5	17.1
Sausage (farmer)	3.9	22.2	27.9	40.4	.....

III. Fuel Value 1500–2000 Calories per Pound

Barley (pearled)	.....	9.8 to 12.9	7.0 to 10.1	0.7 to 1.5	77.3 to 78.1*
Beans (dried)	.....	9.6 to 15.5	19.9 to 26.6	1.4 to 3.1	57.2 to 63.5*
Cake average (except fruit)	.....	19.9	6.3	9.0	63.3
Candy	.....	4.0	.....	.....	96.0
Cheese (Neuchatel)	.....	42.7 to 57.2	15.1 to 22.3	22.3 to 32.5	0.2 to 2.9
Corn meal	.....	8.8 to 17.9	6.7 to 11.6	1.0 to 5.3	68.4 to 80.6*
Corn-starch	.....	10.0	.....	.....	90.0*
Crackers (average)	.....	6.8	10.7	8.8	71.9*
Fat meats	11.7	38.3	13.0	36.8	.....
Gelatin	.....	13.6	84.2	0.1	.....
Ham (smoked, medium fat)	4.5 to 28.4	27.3 to 42.5	10.2 to 21.9	24.5 to 39.9	.....
Infants' and invalids' foods	.....	2.4 to 12.3	2.0 to 22.5	0.3 to 10.9	66.9 to 89.4
Macaroni	.....	7.0 to 12.3	7.9 to 16.6	0.0 to 4.9	67.2 to 78.4*
Oats	.....	7.8	16.5	7.3	66.5*
Peanuts	24.5	6.9	19.5	29.1	18.5
Peas (dried)	.....	6.9 to 15.0	20.4 to 28.0	0.8 to 1.3	58.0 to 67.4*
Pop-corn	.....	4.3	10.7	5.0	78.7
Rice	.....	9.1 to 14.0	5.9 to 11.3	0.1 to 0.7	75.4 to 81.9*
Rye flour	.....	11.9 to 13.6	4.9 to 8.8	0.2 to 1.3	77.6 to 80.2*
Sugar (granulated)	.....	.....	.....	.....	100
Wheat (entire) flour	.....	6.4 to 13.1	12.2 to 14.6	1.5 to 2.1	69.5 to 77.0*
Wheat flour (white bakers')	.....	10.1 to 13.3	10.3 to 14.9	1.9 to 2.0	70.3 to 75.5
Wheat (shredded)	.....	7.2 to 10.7	9.6 to 11.4	1.3 to 1.6	75.0 to 79.7*
Zwieback	.....	5.0 to 7.7	8.6 to 11.7	8.1 to 11.3	72.1 to 74.2

* Including fibre.

IV. Fuel Value 1000–15000 Calories per Pound

Apples (dried)	.....	8.6 to 47.4	1.2 to 2.5	0.1 to 5.0	48.6 to 86.91
Bread (white)	.....	35.3	9.2	1.3	53.1
Corn-bread	.....	28.4 to 48.0	6.5 to 10.1	2.3 to 9.8	40.3 to 54.3
Dates	10.0	13.8	1.9	2.5	70.6
Figs	.....	11.6 to 25.0	2.6 to 5.7	0.3	68.3 to 83.1
Fresh pork (ribs and shoulder)	15.9 to 20.3	40.1 to 43.6	13.7 to 14.5	25.4 to 25.6	.....
Medium fat mutton and beef	14.4 to 27.8	38.0 to 44.9	11.4 to 12.9	19.8 to 31.2	.....
Mince-meat (commercial)	.....	27.7	6.7	1.4	60.2
Mince-meat (home-made)	.....	54.4	4.8	6.7	32.1
Pies	.....	44.9	4.4	9.4	39.2
Prunes (dried)	15.0	19.0	1.8	.....	62.2
Raisins	10.0	13.1	2.3	3.0	68.5
Sandwiches	.....	44.9	10.9	9.0	33.3
Sardines (canned)	5.0	53.6	23.7	12.1	.....
Salt mackerel	22.9	32.5	16.3	17.4	.....

* One Calorie equals 1000 calories.

COMPOSITION OF SOME COMMON FOOD MATERIALS. — *Continued*

V. Fuel Value 500–1000 Calories per Pound

Food-material.	Refuse.	Water.	Nitrogenous Substances	Fat.	Carbohydrates.
	Per cent.	Per cent.	Per cent.	Per cent	Per cent.
Beef (round)	8.5	62.5	19.2	9.2	.....
Beef (sirloin steak)	12.8	54.0	16.5	16.1	.....
Chicken (fowls)	18.0 to 42.7	38.3 to 53.7	11.5 to 16.0	6.9 to 21.5	.....
Cream	.....	74.0	2.5	18.5	4.5
Eggs	11.2	65.5	11.9	9.3	.....
Herring (smoked)	44.4	19.2	20.5	8.8	.....
Meats (lean)	0.5 to 11.3	59.9 to 69.2	18.1 to 21.4	7.8 to 14.2	.....
Olives	19.0	52.4	1.4	21.0	3.5
Salmon (fresh)	23.8 to 35.1	45.0 to 51.2	12.6 to 15.0	6.6 to 9.5	.....
Salmon (canned)	11.7 to 16.9	54.6 to 58.2	18.6 to 20.2	5.6 to 9.8	.....
Tapioca pudding	.....	52.0 to 71.6	2.8 to 4.2	2.3 to 4.8	21.9 to 38.1
Tongue (beef)	9.2 to 55.3	32.4 to 69.2	7.8 to 20.2	0.7 to 15.3	.....
Turkey	17.1 to 32.4	41.1 to 44.7	15.8 to 16.8	5.9 to 25.5	.....
Veal (breast)	15.7 to 25.4	48.5 to 55.7	14.2 to 16.9	9.4 to 12.8	.....

VI. Fuel Value 400–500 Calories per Pound

Beans (canned red kidney)	.....	72.7	7.0	0.2	18.5
Calf's-foot jelly	.....	77.6	4.3	.....	17.4
Salt cod (boneless)	1.6	54.8	27.7	0.3	.....
Succotash (canned)	.....	71.4 to 79.9	2.9 to 4.4	0.7 to 1.7	14.9 to 22.4
Sweet potatoes	20.0	55.2	1.4	0.6	21.9

VII. Fuel Value 300–400 Calories per Pound

Bananas	35.0	48.9	0.8	0.4	14.3
Butter beans	50.0	29.4	4.7	0.3	14.6
Fish (fresh)	25.2 to 46.0	46.1 to 49.1	11.9 to 12.0	1.8 to 5.9	.....
Grapes	25.0	58.0	1.0	1.2	14.4
Hash	.....	80.3	6.0	1.9	9.4
Milk	.....	87.0	3.3	4.0	5.0
Potatoes	20.0	62.6	1.8	0.1	14.7

VIII. Fuel Value 200–300 Calories per Pound

Apples	25.0	63.3	0.3	0.3	10.8
Chicken (broilers)	31.4 to 55.1	44.6 to 52.4	9.0 to 15.7	1.1 to 1.8	.....
Cranberries	.....	87.6 to 89.5	0.4 to 0.5	0.4 to 0.9	9.3 to 10.9
Onions	10.0	78.9	1.4	0.3	8.9
Oysters (solid)	.....	82.2 to 92.4	4.5 to 7.3	0.5 to 1.8	1.5 to 6.2
Parsnips	20.0	66.4	1.3	0.4	10.8
Pears	10.0	76.2	0.5	0.4	12.7

IX. Fuel Value 100–200 Calories per Pound

Beets	20.0	70.0	1.3	0.1	7.7
Cabbage	15.0	77.7	1.4	0.2	4.8
Carrots	20.0	70.6	0.9	0.2	7.4
Green corn	61.0	29.4	1.2	0.4	7.7
Lemons	30.0	62.5	0.7	0.5	5.9
Oranges	27.0	63.4	0.6	0.1	8.5
Soups (canned)	.....	91.0 to 92.8	2.9 to 5.0	0.2 to 0.8	0.6 to 5.7
Spinach	.....	91.6 to 92.8	1.8 to 2.4	0.2 to 0.5	3.1 to 3.4
Squash	50.0	44.2	0.7	0.2	4.5
Tomatoes (canned)	.....	92.5 to 97.9	0.3 to 1.7	0.1 to 0.3	1.4 to 8.1

X. Fuel Value 10–100 Calories per Pound

Asparagus	.....	94.0	1.8	0.2	3.3
Bouillon (canned)	.....	96.5 to 96.7	1.7 to 2.6	0.0 to 0.2	0.1 to 0.3
Celery	20.0	75.6	0.9	0.1	2.6
Cucumbers	15.0	81.1	0.7	0.2	2.6
Watermelons	59.4	37.3	0.2	0.1	2.7

complete nutrition. The nutritive ratio, or the proportion of nitrogenous to non-nitrogenous food, must be maintained in the proportion of 1 to 3, or at least 1 to 5.

The preceding table of one hundred common food-materials is arranged in the order of calorific or energy-giving power, but in considering the food value of any one substance its nitrogen content must also be considered, and such combinations made as will yield the requisite elements for a well-balanced ration.

From even a cursory examination of the table it will be seen how widely some of the foodstuffs differ under differing conditions of soil moisture, fertilization in the case of plants, and of fatness or leanness in animals, of method of preparation or of combination in cooked foods.

Therefore examinations of materials are imperative if there is to be any basis of calculation. In an institution where, for instance, flour forms two-thirds of the daily ration, if it contains the lowest per cent of nitrogen it may not furnish sufficient proteid for a well-balanced ration, or if the meat used is very lean there may not be fat enough for the best nutrition.

The great variation in the proportion of water leads to many surprises, and the amount of unedible material is to be considered. The uneducated provider buys oysters under the impression that he is furnishing food of high value, and does not distinguish between potatoes and rice.

In the present state of our knowledge, the best use to which we can put such tables and analyses is as a check against gross errors of diet, which are found with alarming frequency especially among children and students, those who can least afford to make them. References will be found in the Bibliography to works for further study along these lines.

Dietaries. — A dietary is simply a known amount of food of known composition per person per day, week, or month.

What is called a standard dietary is such a combination of food-materials as shall furnish the amounts held to be necessary. The following are examples of such standard dietaries:

Approximate amounts required daily by	Nitrogenous, grams.	Fats, grams.	Carbohydrates, grams.	Calories.
Child of 6–9	62	45	200	1593
Child of 9–14	78	45	281	1890
Adult at rest	100	75	380	2665
Adult at moderate work	100	90	450	3092
Adult at hard work	125	125	500	3725

(In feeding experiments from 10 to 20 per cent more must be allowed for waste and indigestibility.)

From the table on page 118 may be selected such food as will give the required quantities in variety enough to suit any taste. That which the table cannot give is the per cent of each which, under any given condition, will be utilized by the person fed. The strength of the digestive juices, exercise, fresh air, the cooking, the mixing of the foods, the habits of mind as to food, the customs of the family, all influence this utilization, so that other means must be restored to in order to gain an idea of what is practicable. This is done by taking account of the food of persons free to choose; of those in different countries, in different circumstances, and using a great variety of materials. Since Voit made his standard dietary in 1870, many hundreds, at least, have been so gathered in the United States alone — more than two hundred since 1886. All the information thus gained goes to confirm the theoretical standard, and also to show how much depends upon suitable preparation and combination. These last two things help each other.

As food is ordinarily prepared, about 10 per cent must be deducted for indigestibility in a customary mixed diet, and about 10 per cent more for the refuse or waste of food as purchased, so that of the total pounds of meat, vegetables, and groceries some 20 per cent is of no final service in the body. It is immaterial whether this amount is subtracted from the final calculation or whether the higher figures be taken, that is, whether 125 grams of proteid as purchased or 100 grams final utility is used. There will be an unknown limit in either case. According to late experiments 100 grams of proteid is high.

The waste of fats is less in proportion as the dietary is a restricted one.

Knowledge of Food Values Necessary. — The most serious aspect of the food question is that the taking of it is voluntary, not, like air, a necessity beyond control, and that the most fantastic ideas are allowed to rule. The day-laborer is in little danger, since his food demand is made strong by out-of-door exercise; but the student who shuts himself up in hot, close rooms, and who does not look upon food as his capital, but only as a disagreeable task or an amusement, is in great danger, as is he who, having heard that one can live on a few cents a day, proceeds to try it without knowledge, and suffers a loss of efficiency for years or for all his life.

It is not nearly so difficult to acquire a working knowledge of food values as of whist or golf, so that on entering a restaurant a suitable menu may be made up within one's allowance. It is only necessary to correct prevailing impressions and reinforce one's experience.

Figs, dates, raisins and prunes are apt to be regarded as luxuries instead of as rich food-substances of a most digestible kind when freed from skin and seed. Nuts are a much neglected form of wholesome food, admirably suited to a winter table from their richness in fat, and also furnishing muscular energy, as is seen in the agile squirrel, and is proved by many human examples. With nuts, however, must be taken fruits or other bulky foods, to balance the concentration. The somewhat compact and oily substance must be finely divided and freed from its astringent skin.

In distinction from these rich foodstuffs, we find oranges, apples, etc.; the usual garden vegetables, asparagus, lettuce, etc., which while they fill an important place in the dietary, add little directly to the energy of the body and need not be considered except as, by their flavor or æsthetic stimulus, they add to the efficiency of the rest.

The foods which furnish the greatest nutrition for the least money are such materials as corn meal, wheat flour, milk,

beans, cheese and sugar. The expensive cuts of meat, high-priced breakfast cereals and the like, add but little to the nutritive value but greatly increase the cost of living. A meal of lettuce dressed with oil, eaten with bread and cheese, fulfils all the requirements of nutrition, and may cost five cents. The same food value from sweet breads, grape-fruit, etc., might cost a dollar. Incorrect ideas in regard to food values, and prejudice inherited or acquired against certain foods, have too often resulted in excluding wholesome and nutritious articles from the dietary and decreasing thereby the efficiency of the human machine.

CHAPTER VIII

THE PROBLEM OF SAFE FOOD. ADULTERATION AND SOPHISTICATION

ADULTERATION grows largely, if not almost entirely, from excessive competition. Nearly every article of common food has been found at one time or another to be adulterated, yet manufacturers testify that they willingly would stop this addition of foreign material if they could be sure that their competitors would stop also. Other causes there are also: demands for goods out of season; for perishable products which must come many miles; the failure of the supply of a given substance to meet a continuing demand; all of these lead to adulteration, imitation and substitution.

To many people otherwise intelligent, the term *adulterated* food is synonymous with *poisoned* food. With others, thanks to alarming newspaper articles, not wholly disinterested, the general impression is far beyond the reality. It is not necessary to use poisonous or even deleterious material; it needs only to mix with the food material some substance cheaper but harmless, to make some change in the outward appearance of the article so that people shall not recognize the familiar substance, and then to herald far and wide the discovery of a new process by which the food value is greatly enhanced. "Things are not what they seem" is nowhere more true than in the case of foods.

Definition of Adulteration. — To adulterate is "to *debase*," "to make impure by an admixture of baser materials." The word "adulterated" refers to any food to which any foreign substance, not a proper portion of the food, has been added. It does not matter whether the added material is of greater value than the food itself. The addition of coffee to cereal or substitute coffees,

is properly held to be an adulteration. Deterioration should not be mistaken for adulteration. People who are not wholly familiar with the appearance of a food or the chemical and physical changes which it may undergo, think that if it does not taste just right or look just right that it must be adulterated. Appearance has slight relation to the purity of the article in these days of paint, polish and powder.

Some forms of adulteration are more properly described under the head of *misbranding*, that is, referring to foods incorrectly described by the label. While the significance is not exactly the same as that of the word adulterated, yet the two may sometimes be applied to the same product. For instance, the addition of starch to sausage to conceal the use of excessive amounts of water and of fat constitutes an adulteration, which would not be the case if the article were properly branded to show the presence of the added "filler."

To adulterate the coin of the realm or the liquor of the bar with a baser metal or an imitation whisky is a heinous offence. So is the mixture of milk with the baser article, water, which thereby lowers its food value. But the "wretched sophistry" which obscures the nature of things on a package of prepared food misleads more persons and inflicts more injury upon the community than the other, yet goes unrebuked. The most barefaced assertions are printed in magazines, and "pure-food shows" only whet the appetite for something new.

Legal Definition of Adulteration and Misbranding. — In the Federal Pure Food Law, commonly known as the Food and Drugs Act of June 30, 1906, adulteration and misbranding are thus defined:

Sec. 7. That for the purposes of this Act an article shall be deemed to be adulterated:

In the case of food:

First. If any substance has been mixed and packed with it so as to reduce or lower or injuriously affect its quality or strength.

Second. If any substance has been substituted wholly or in part for the article.

Third. If any valuable constituent of the article has been wholly or in part abstracted.

Fourth. If it be mixed, colored, powdered, coated, or stained in a manner whereby damage or inferiority is concealed.

Fifth. If it contains any added poisonous or other added deleterious ingredient which may render such articles injurious to health: *Provided*, That when in the preparation of food products for shipment they are preserved by any external application applied in such manner that the preservative is necessarily removed mechanically, or by maceration in water, or otherwise, and directions for the removal of said preservative shall be printed on the covering or the package, the provisions of this Act shall be construed as applying only when said products are ready for consumption.

Sixth. If it consists in whole or in part of a filthy, decomposed, or putrid animal or vegetable substance, or any portion of an animal unfit for food, whether manufactured or not, or if it is the product of a diseased animal, or one that has died otherwise than by slaughter.

SEC. 8. That the term "misbranded," as used herein, shall apply to all drugs, or articles of food, or articles which enter into the composition of food, the package or label of which shall bear any statement, design, or device regarding such article, or the ingredients or substances contained therein which shall be false or misleading in any particular, and to any food or drug product which is falsely branded as to the State, Territory, or country in which it is manufactured or produced.

That for the purposes of this Act an article shall also be deemed to be misbranded:

In the case of food:

First. If it be an imitation of or offered for sale under the distinctive name of another article.

Second. If it be labeled or branded so as to deceive or mislead the purchaser, or purport to be a foreign product when not so, or if the contents of the package as originally put up shall have been removed, in whole or in part, and other contents shall have

been placed in such package, or if it fail to bear a statement on the label of the quantity or proportion of any morphine, opium, cocaine, heroin, alpha or beta eucaine, chloroform, cannabis indica, chloral hydrate, or acetanilide, or any derivative or preparation of any such substances contained therein.

Third. If in package form, and the contents are stated in terms of weight or measure, they are not plainly and correctly stated on the outside of the package.

Fourth. If the package containing it or its label shall bear any statement, design, or device regarding the ingredients or the substances contained therein, which statement, design, or device shall be false or misleading in any particular: *Provided*, That an article of food which does not contain any added poisonous or deleterious ingredients shall not be deemed to be adulterated or misbranded in the following cases:

First. In the case of mixtures or compounds which may be now or from time to time hereafter known as articles of food, under their own distinctive names, and not an imitation of or offered for sale under the distinctive name of another article, if the name be accompanied on the same label or brand with a statement of the place where said article has been manufactured or produced.

Second. In the case of articles labeled, branded, or tagged so as to plainly indicate that they are compounds, imitations, or blends, and the word "compound," "imitation," or "blend," as the case may be, is plainly stated on the package in which it is offered for sale: *Provided*, That the term blend as used herein shall be construed to mean a mixture of like substances, not excluding harmless coloring or flavoring ingredients used for the purpose of coloring and flavoring only: *And provided further*, That nothing in this act shall be construed as requiring or compelling proprietors or manufacturers of proprietary foods which contain no unwholesome added ingredient to disclose their trade formulas, except in so far as the provisions of this act may require to secure freedom from adulteration or misbranding.

Extent of Adulteration. — In any discussion of the extent to which adulterated foods are sold it must be borne in mind that the adulterated articles make up only a relatively small proportion of the food that actually passes over the counter. Flour, for example, is seldom adulterated; pepper, mustard and vanilla extract often are. For one pound of these substances sold, 1000 pounds or more of flour go out from the store. Figures given in official reports of food inspection do not represent the case exactly, because the inspectors are trained men, and purchase samples of those lines of goods which experience has shown them to be most likely to be adulterated. Brands of foods which they have reason to believe are pure they do not sample. Estimated on the total quantity sold, it is doubtful if more than 5 to 10 per cent of the food sold is adulterated in any way, and these figures would undoubtedly be much too high for those states in which there is a well-enforced system of food inspection.

Character of Adulteration. — Much of the present propaganda in the interests of pure food and the movement for the protection of the consumer can be summed up in three words: "An Honest Label." In many cases an accurate and true statement of the contents of the can or package is the only protection needed by the consumer, and is fully as efficient as well as much cheaper than prosecutions or restrictive measures. Many of the terms used on food packages deceive only the ignorant purchaser. "Strictly pure" is a well-understood trade term, with a meaning known to the initiated; the words "Home-Made" may cover some of the most highly developed products of synthetic organic chemistry.

The cases in which the adulteration is of a character deleterious to health are fortunately few. The use of canned goods brings certain dangers in the dissolved metals from the cans or from the solder, also from a careless habit of allowing foods to stand in the opened tins. The liking for bright green pickles and peas leads to coloration by copper salts.

So rapidly do new substances come upon the market that it is of little use to put into a general text-book definite statements of

the quality of many foods. A baking-powder or a spice which is honestly made to-day may next week pass into the hands of unscrupulous dealers who please the public and thereby salve their consciences.

To furnish what the people *think* they want has been the rule from the days of an earlier generation of grocers, who divided a barrel of cooking-soda in halves and set one-half on one side of the store for "saleratus" and the other on the opposite side for soda, so that there should be no suspicion in the mind of the customer that the packages came from the same barrel, and yet each might satisfy his individual preference.

Names that have passed down from a former generation as being above reproach are now found to cover adulterated goods. The trademark has passed into other and less scrupulous hands, and the new owners do not hesitate to trade upon the reputation earned by their predecessors. There are, however, several phases of the subject that should be briefly mentioned.

Breakfast Foods. — The craving for something new to stimulate a jaded appetite already spoiled by endless variety and bad combinations has led to the manufacture of a cereal preparation for nearly every day in the year, regarding some of which the statement is made that they are "predigested." No better commentary on the laziness or wilful ignorance of American providers could be made than this. Little do the people know about wheat or cooking if they suppose that grain can be changed by manipulation in any kind of machine so as to give greater food value than was contained in the grain. While it is true that some of these preparations are far better than the half-cooked grains found on so many tables, the fact remains that it is the cook and not the substance which is poor. The false statements on food packages of all kinds are so absurd that they would defeat their own purpose were they viewed in the light of common sense. It is not always best to have food which is too easily digested.

A predigested food is quickly absorbed into the circulation, and hence a small quantity causes a sensation of fulness and satisfaction, which, however, soon passes away and a faintness results.

This is especially true of the sugars and dextrins. Frequent meals should go with easily absorbed foods. The rapid digestion is the cause of much pernicious eating of sweets between meals, which satisfies the appetite for the time being and prevents substantial quantities of other foods being taken at the time they are offered.

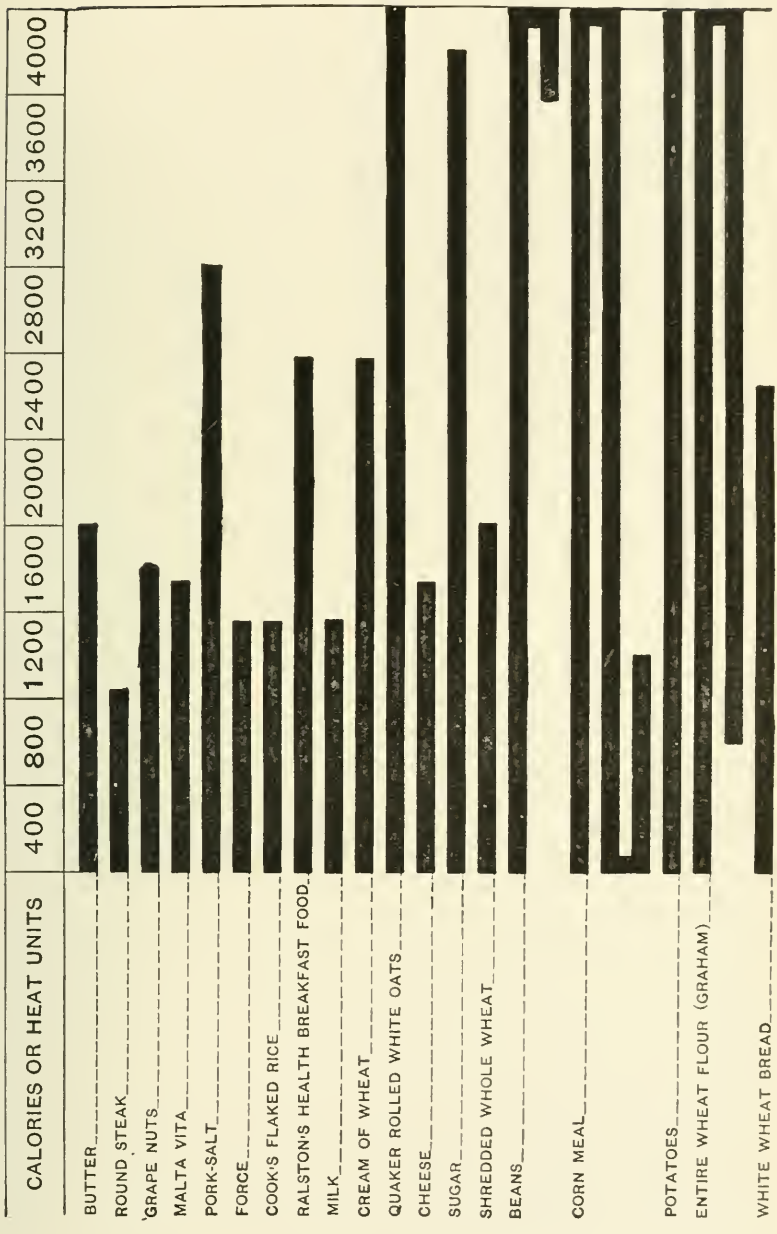
From a study of analyses of a large number of foods the following conclusions are drawn by F. W. Robison: *

1. The breakfast foods are legitimate and valuable foods.
2. Predigestion has been carried on in the majority of them to a limited degree only.
3. The price for which they are sold is as a rule excessive and not in keeping with their nutritive values.
4. They contain, as a rule, considerable fibre which, while probably rendering them less digestible, at the same time, may render them more wholesome to the average person.
5. The claims made for many of them are not warranted by the facts.
6. The claim that they are far more nutritious than the wheat and grains from which they are made is not substantiated.
7. They are palatable as a rule and pleasing to the eye.
8. The digestibility of these products as compared with highly milled goods, while probably favorable to the latter, does not give due credit to the former, because of the healthful influence of the fibre and mineral matter in the breakfast foods.
9. Rolled oats or oatmeal as a source of protein and of fuel is ahead of the wheat preparations, excepting of course the special gluten foods, which are manifestly in a different class.

In general, the cost of these foods is low if they are considered merely as confections to please the taste, but they are expensive foods regarded as substitutes for the ordinary cereal products.

This is well shown in the following table in which the fuel value of breakfast foods and other common food products obtained for a given sum is graphically compared.

* Mich. Agr. Expt. Sta., *Bull.*, 211 (1904).


CALORIES OR HEAT UNITS
400
800
1200
1600
2000
2400
2800
3200
3600
4000
BUTTER
ROUND STEAK
GRAPE NUTS
MALTA VITA
PORK-SALT
FORCE
COOK'S FLAKED RICE
RALSTON'S HEALTH BREAKFAST FOOD
MILK
CREAM OF WHEAT
QUAKER ROLLED WHITE OATS
CHEESE
SUGAR
SHREDDED WHOLE WHEAT
BEANS
CORN MEAL
POTATOES
ENTIRE WHEAT FLOUR (GRAHAM)
WHITE WHEAT BREAD

Colors and Preservatives in Food. — For many years such substances as alcohol, vinegar, sugar, salt, and the like, have been used to preserve food. Such materials are commonly held to be harmless to persons of sound digestion if used in moderate amounts. Within recent years, however, there has been a constantly increasing tendency toward the use in food products of such powerful antiseptics as formaldehyde, salicylic and benzoic acids and their salts, and boric acid. An important distinction to be borne in mind between this class of preservatives and those first named is that the former when used in food in quantity sufficient to preserve it make their presence known to the consumer by either their taste or odor. With the chemical preservatives, however, an intimation of their presence is conveyed to the consumer only by a statement on the label. It is the general feeling among those engaged in the enforcement of the food laws that the common use of these preservatives should be forbidden, or that they should be allowed only under certain definite restrictions. The question is not one of their possible harmful effect only, although it cannot be successfully denied that their unrestricted use would lead to grave danger to health, especially in the case of invalids and children, or those with various degrees of digestive efficiency. It seems reasonable to infer that the processes of digestion, being largely the result of bacterial and enzymic action, will be retarded or interfered with to a greater or less extent by substances which inhibit bacterial action in food.

There is, however, another reason for objecting to the use of chemical preservatives. By their use much food that is unwholesome and unfit for consumption can be, and is, placed upon the market with no warning to the consumer. "The man who adds formaldehyde to his milk takes down the danger signal, but does not remove the danger."

Similarly, objections can be made to the use of coal-tar colors in foods. There are hundreds of food packages which would never leave the grocers' shelves were it not for the fact that by the use of artificial color their true composition and the actual

nature of the materials from which they are made is hidden. Apart from any question as to the harmfulness of these dyes there is ample reason for their use being strictly regulated by official action, in that their use except under such supervision allows the manufacturer to sell inferior articles under the appearance of standard foods; it permits the customer to be misled as to the strength and purity of the product that he buys; the age and past history of the product may be made a sealed book; finally, by the use of coloring, an unwholesome and improper food may be put upon the market.

Summary. — The chief dangers in food are from wrong proportions of proteid, fat, and carbohydrates, from fermentable and irritating decompositions, from bad methods of cooking and unsuitable combinations, from transmission of micro-organisms either by exposure to dust or by contact with filthy hands or vessels, to a favorable medium for the growth of pathogenic germs, from unsuitable food scientifically disguised.

From this hasty survey it will be seen how little danger to health is incurred if only reasonable care is taken and if the always doubtful articles are avoided.

Take, for instance, that most commonly adulterated class, spices. Who will say that it may not be better to eat corn and buckwheat and ground peas than pure pepper? Rice is certainly a more wholesome food than ginger, and starch than soda. Glucose is even more easily absorbed than cane-sugar. These are cases of frauds on the pockets, but possibly blessings in disguise for the stomachs. When any community is so ignorant as to permit of such glaring cases of adulteration as coal-tar dyes in food, and gypsum in cream of tartar, they deserve to suffer. It is knowledge on the part of each intelligent citizen which will mend matters, even if it is only that kind of empirical knowledge that one is forced to learn in relation to electricity and steam in order to live in a modern house.

This knowledge is now easily obtained through the city, state and governmental laboratories, and their publications are accessible to all who can read and write. There is therefore no excuse

for general ignorance and credulity as to trade preparations of foods, any more than for the degrading habit of purchasing patent medicines to remedy the ills caused by the misuse of food. Both together form the saddest commentary on human weakness and lack of rational thought.

CHAPTER IX

ANALYTICAL METHODS

In the discussion of the methods employed for the examination of food-materials, only a few typical substances have been considered, and the processes given are such as to bring into prominence the scientific aspect rather than the technical detail of the subject; at the same time it is hoped that a sufficient variety of methods is given to enable the student to gain considerable experience in the necessarily short time which can be alloted to the subject.

Both on account of its importance as a food-material and on account of its availability for the various tests, milk has been chosen as a type of animal food; moreover, it may be made to serve as an excellent example of the changes to which food-materials are liable through the growth of the micro-organisms. The analysis of milk includes determinations of specific gravity, water, or total solids, ash, fat, proteids and sugar, the separation of casein and albumin, and the detection of preservatives, coloring matters, and added water.

The breakfast cereals are taken as typical of vegetable foods. The examination which may be made of this class includes the determination of moisture, ash, fat, nitrogen and proteids, starch, cellulose, and the products of peptonization and saccharification.

The nature and composition of the various fats and oils is briefly illustrated by the examination of butter and the determination of the principal "constants" of the butter-fat.

The results of fermentation are illustrated by the determination of alcohol in beer, wine, meat extracts, patent medicines and "temperance drinks," flavoring essences and the like. The determination of the relative proportion of volatile and

fixed acids, of the saccharine products of malting, and of volatile oils or flavoring principles, is also instructive.

A more elaborate discussion of the methods used in food analysis and of the interpretation of results will be found in the larger works upon the subject. As reference books for the use of the student in the laboratory, the following, in the author's experience, have been found especially helpful: Leach: Food Inspection and Analysis; Sherman: Organic Analysis; Rolfe: The Polariscope in the Laboratory; Bulletin 107, Bureau of Chemistry.

MILK

Milk is a food material of somewhat complex and variable composition but can be described as essentially an aqueous solution of milk sugar, mineral salts and soluble albumin containing suspended globules of fat and partially dissolved casein.

General Composition. — In approximate figures the average percentage composition of milk may be stated:

	Per cent
Total solids	12.8
Fat	3.8
Protein	3.6
Ash	0.7
Milk sugar	4.7
Solids not fat	9.0

From these figures there may be in normal milk quite decided variations and figures have been reported which differ widely from them, some of the discrepancies of the older analyses being undoubtedly due to the imperfect methods of analysis employed.

Lythgoe * states that all milk completely drawn from healthy cows will fall between the following limits:

	Extreme limits, per cent.	Usual limits, per cent.	Herd milk, per cent.
Total solids	10.0–17.0	10.5–16.0	11.8–15.0
Fat	2.2– 9.0	2.8– 7.0	3.2– 6.0
Protein	2.1– 8.5	2.5– 4.5	2.5– 4.0
Ash	0.6– 0.9	0.7– 0.8	0.7– 0.8
Milk sugar	4.0– 6.0	4.2– 5.5	4.3– 5.3
Solids not fat	7.5–11.0	7.7–10.0	8.0– 9.5

* *Bull. Mass. State Bd. Health*, 1910, p. 419.

Variations in Composition. — Besides variations in composition which may be due to individual cows there are also certain well-established differences due to environment or to racial influences. Among the more important of these are:

(1) *The Breed of the Cow.* — Some breeds yield quantity, others quality. The Jersey and Guernsey cattle, for instance, give comparatively small quantities of milk rich in fat; the Holstein cows, on the other hand, yield much larger amounts of milk of decidedly lower solids and fat content. These differences are well summarized in the following table based on data collected by the Massachusetts Board of Health.*

Breed.	Specific gravity.	Total solids, per cent.	Fat, per cent.	Protein, per cent.	Ash, per cent.	Solids not fat, per cent.	Milk sugar, per cent.
Jersey	1.034	14.57	5.40	3.54	0.78	9.17	4.85
Guernsey	1.034	14.40	5.00	3.77	0.77	9.40	4.86
Ayrshire	1.032	12.57	4.00	2.90	0.77	8.57	4.90
Dutch Belt	1.032	12.03	3.60	2.62	0.68	8.43	5.00
Holstein	1.032	11.96	3.35	2.99	0.69	8.61	4.89

If individual differences are eliminated and only fully drawn mixed milk from herds is considered, the variation due to breed is the factor of the greatest influence in permanently affecting the composition of milk.

(2) *The Time of Year.* — The poorest milk is produced during the spring and early summer months, the richest during the seasons of autumn and early winter, when the cattle are getting a smaller proportion of green feed. This difference is clearly shown in the following table † which gives the seasonal average for 16 years:

	Total solids, per cent.	Fat, per cent.	Solids not fat, per cent.
Nov.–Jan.	13.04	4.11	8.93
Feb.–Apr.	12.72	3.88	8.84
May–Aug.	12.66	3.89	8.77
Oct.–Nov.	13.03	4.25	8.78

* *Bur. of Chem., Bull.* **132,** p. 129.

† Richmond: Dairy Chemistry, p. 126.

This variation in composition of milk between the pasture-fed and the stall-fed season has in the past received legal recognition in the fixing of milk standards. In Massachusetts for many years the legal standard for total solids was set at 13 per cent in the winter months and at 12 per cent in the summer season.

(3) *Time of Day.* — Milk which has been drawn in the evening is nearly always richer in fat than the morning milk as shown in the following averages:

	Specific gravity.	Total solids.	Fat.
Morning milk	1.0322	12.53	3.63
Evening milk	1.0318	12.94	4.04

(4) *"Fore" milk vs. "strippings."* — If different portions of the whole quantity of milk obtained at a single milking are examined separately they will be found to show marked differences in fat content, especially as between the first and last portions. The other constituents of the milk do not vary so greatly as the fat. The first portions of milk, the "fore" milk, contain much less fat than do the last portions or "strippings." The following figures, due to Van Slyke, illustrate this point:

	Per cent of fat in milk.		
	Cow 1.	Cow 2.	Cow 3.
First portion drawn	0.90	1.60	1.60
Second portion drawn	2.60	3.20	3.25
Third portion drawn	5.35	4.10	5.00
Fourth portion drawn (strippings)	9.80	8.10	8.30

This difference in composition is explained by the separation of the milk while in the udder of the cow, cream rising to the top just as would happen if the milk stood in a vessel, hence being drawn last. Dishonest dairymen have in the past taken advantage of this fact in adulteration cases, by having the cows *partially* milked in the presence of unsuspecting witnesses, the resulting "known purity" milk being thus largely "fore" milk.

In general it will be found that to whatever causes the variations noted in the composition of milk are due, the differences are shown much more in the fat than in any other constituent. The protein is also variable, although to a somewhat less extent, and the milk sugar and ash are much more nearly constant.

METHODS OF ANALYSIS

Preparation of the Sample. — Since the cream will rise on a sample of milk sufficiently in five minutes to destroy the uniformity of the sample, great care must be used in taking a portion for analysis to ensure that it represents a fair average of the milk. The best way is to pour the milk from the containing vessel into another and back again several times, or if this is impracticable it should be thoroughly stirred before being sampled. If the analytical sample has stood for any appreciable time it should be mixed by pouring back and forth before a portion is removed to test, otherwise concordant results cannot be obtained. Do not shake the sample since this tends toward a separation of the fat.

Specific Gravity. — This is usually taken with a special form of hydrometer, known as a *lactometer*. The Quevenne lactometer has a scale graduated into 25 equal parts, extending from 15 to 40, corresponding to specific gravities from 1.015 to 1.040. The best form of instrument is that provided with a thermometer.

The lactometer is graduated to give correct results at 60° F. (15.6° C.) and the reading should be made at approximately that temperature, between 55 and 65 degrees, and then corrected to standard temperature. This may be done by adding 0.1 to the reading for each degree F. above 60° F., or subtracting 0.1 for each degree F. below 60° F. If the temperature is read in Centigrade degrees the correction may be made by the table on page 216.

The New York Board of Health lactometer has a scale reading 0 in water, and 100 in milk with a specific gravity of 1.029, which is taken as the lowest limit for pure milk. The instrument is used in the same manner as the Quevenne lactometer and the

readings can readily be converted into degrees of the latter instrument.

Notes. — The specific gravity of milk fat is about 0.93; of the solids not fat approximately 1.5. The specific gravity of the milk itself is thus a function of the two; the former lowers it, the latter increases it. As would be expected from the variable composition of milk, the specific gravity is also a variable. The values for normal milk from a herd, however, will usually fall between 1.030 and 1.034.

Taken by itself the specific gravity is of little value in showing adulteration. The addition of water lowers the specific gravity of milk; the removal of cream raises it, this being the lighter portion of the milk. It is therefore theoretically possible by skilful manipulation to both skim and water a sample and still have its specific gravity correspond to that of normal milk. Such a sample would, however, be readily recognized by one familiar with the appearance of the genuine product.

The lactometer reading is of value in rapid analysis of milk for calculating the solids in connection with the Babcock method of fat determination (see page 148).

Total Solids. — Use a platinum dish having a flat bottom about $2\frac{1}{2}$ inches in diameter. Ignite and weigh the dish accurately, then add about 5.1 grams to the weights on the balance-pan. With a pipette deliver 5 c.c. of the well-mixed milk into the dish and weigh the whole as rapidly as possible to the nearest milligram. Evaporate the milk to dryness on the water-bath and then dry it in the oven at 100° C. to constant weight. Three hours drying is usually sufficient.

Notes. — It is important that the milk should be dried in a thin layer, so that the removal of the water shall take place as quickly as possible. Under these conditions the residue obtained is nearly white, but if the process be prolonged, it may have a brownish color from the caramelization of the sugar.

If it is not desired to determine ash on the same weighed portion as used for the solids, lead foil dishes or tin blacking box covers may be used instead of platinum dishes.

Ash. — Ignite the platinum dish containing the residue from

the preceding determination at a low red heat until the ash is white or of a uniform light gray color. This may be done in a muffle furnace at a temperature not exceeding about 600° C., or over a burner carefully regulated so that the dish is nowhere heated above the slightest visible redness.

The ash, after weighing, may be tested for boric acid or carbonates as described on page 154.

Fat. — (a) **Adams' Paper Coil Method.** Roll a strip of fat-free blotting paper * about 22 inches long and $2\frac{1}{2}$ inches wide, into a loose coil and fasten it by a bit of wire. Hold the coil in one hand and slowly run on to the upper end of it exactly 5 c.c. of milk from a pipette. If preferred, about 5 grams of milk may be weighed quickly in a small beaker, and one end of the coil introduced so as to absorb the milk, care being taken to absorb it as nearly completely as possible. The beaker is then quickly re-weighed.

Place the coil, after charging with the milk, dry end downward, in the water oven and dry it for two hours, then extract it for at least two hours in a Soxhlet extractor as shown in Fig. 11. Use about 100 c.c. of either petroleum ether or anhydrous ethyl ether and weigh the flask to the nearest milligram. At the end of this time disconnect the apparatus when the extractor is nearly full of ether, thus recovering a large portion of the solvent, and evaporate the remainder (*away from a flame*), conveniently by the electric heater, using suction. Dry the fat to constant weight in the water-oven. In

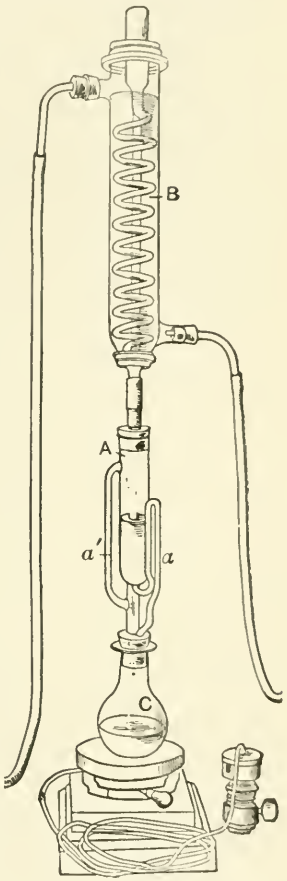


FIG. 11.

* Schleicher and Schüll make suitable strips which can be obtained from dealers in chemical supplies, or the strips may be previously prepared in the laboratory from thick filter paper and extracted with ether before using.

drying the extracted fat it may be heated for two hours the first time, then in one hour periods until the loss of weight is not over a milligram.

Notes. — The only part of the method due to Adams is the drying of the milk on porous paper. This is, however, of great importance since the absorbent paper exercises a selective action on the constituents of milk so that the fat is left on the surface of the paper, mixed with only about one-third of the non-fatty solids, and hence is more easily extracted; further, owing to the greatly increased surface exposed, the extraction of the fat is practically complete in a comparatively short time.

Ethyl ether is the solvent commonly employed but care should be taken that it is anhydrous, otherwise small amounts of milk sugar will be extracted. For this reason petroleum ether is to be preferred as a solvent, although its action is considerably slower than that of the other.

The Adams method is probably the most accurate for fat determination in milk, but in actual practice is not used so much as the more rapid centrifugal methods.

(b) **Babcock Method.** — Measure 17.6 c.c. of the milk from a pipette into the graduated test bottle; add 17.5 c.c. of sulphuric acid (sp. gr. = 1.825) pouring it in slowly so as to form a layer beneath the milk. After the acid has thus been added to all the bottles mix the milk and acid thoroughly by a rotary motion, avoiding the spurting of the liquid into the neck of the bottle. Place the bottles in opposite pockets of the centrifuge in even numbers and whirl them for five minutes at the proper speed. The correct speed varies from 1000 revolutions per minute for a 10-inch wheel to 700 for one of 24 inches diameter. Then remove the bottles and add hot water up to the necks, after which whirl them again for one minute. Again add hot water until the fat rises nearly to the top of the graduations. Whirl again for one minute. Then measure the length of the column of fat by a pair of dividers, the points being placed at the extreme limits of the column, the fat being kept warm, if necessary, by standing the bottles in water at 60° C. If now one point of the

dividers is placed at the 0 mark of the scale on the bottle used, the other will indicate the per cent of fat in the milk.

Notes. — Methods based on centrifugal separation of the fat, of which the Babcock method is the pioneer, are by far the most rapid and convenient for general use. They have practically replaced the more tedious extraction methods and are universally employed in creameries and milk depots.

When the acid and milk are mixed the mixture becomes hot and turns dark colored on account of the charring of the milk sugar. The casein is first precipitated and then dissolved. The retarding effect of the milk serum solids being thus eliminated, the fat globules are free to collect in a mass.

The fat obtained should be of a clear, golden yellow color, and distinctly separated from the acid solution beneath it. If the fat is light-colored or whitish, often with a layer of white particles beneath it, it generally indicates that the acid is too weak or that the milk was too cold when the acid was added. A dark-colored fat with a sub-stratum of black particles indicates that the acid is too strong. The best results will be obtained by the use of acid of the strength noted above.

The capacity of the graduated neck of the bottle between the 0 and 10 marks is 2 c.c. The specific gravity of warm milk fat is 0.9, hence 2 c.c. will weigh 1.8 grams or one-tenth of the weight of 17.6 c.c. of milk (approximately 18 grams). The measurement of the extreme limits of the column of fat, rather than to the upper meniscus, is to correct for the small amount of fat, 0.1 to 0.2 per cent, that remains in the acid solution.

Milk which has been preserved with formaldehyde usually requires a longer time and more vigorous shaking to dissolve the curd, on account of the hardening action of this preservative on the coagulated casein. It is often advantageous to stand the bottles in water at 60° C. for a time before whirling. Samples containing formaldehyde will usually give a violet color when the acid is added to the milk.

(c) **Gottlieb Method.*** — With a pipette place 5 c.c. of milk in a 50-c.c. glass stoppered cylinder and add the following re-

* Röse: *Z. angew. Chem., 1888,* 100; Gottlieb: *Landw. Vers. Stat., 1892,* 6.

agents, being careful to add them in the order given and to shake the stoppered cylinder thoroughly after the addition of each reagent: 1 c.c. of ammonia (sp. gr. = 0.96), 5 c.c. of alcohol, 12.5 c.c. of ethyl ether, and 12.5 c.c. of petroleum ether. Let the cylinder stand until the lower layer is free from bubbles — several hours if necessary. Transfer the upper layer to a tared flask by means of an arrangement similar to a wash-bottle, as shown in Figure 12. Adjust the sliding tube until the end rests just above the junction of the two layers, then by gently blowing force out the upper layer into the flask. Repeat the extraction, using 10 c.c. each of ethyl ether and petroleum ether and blowing it off into the flask as before. Distill off the solvent and dry the residual fat to constant weight in the water oven. Dissolve the weighed fat in a little petroleum ether. If a residue is found, due to a trace of the aqueous layer which was blown off with the ether, wash it several times in the flask by careful decantation with petroleum ether. Finally dry and weigh the flask and residue and deduct from the previous weight. The difference is the weight of purified fat.

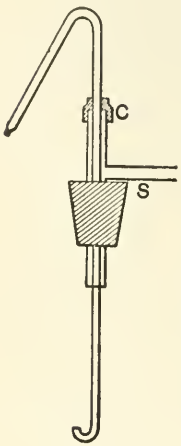


FIG. 12.

Notes. — All of the successful methods for determining the fat by direct extraction from the milk itself involve the complete or partial solution of the casein. In the Gottlieb method the casein, precipitated from the milk in very finely divided form by the alcohol, is dissolved by the ammonia. The fat is dissolved by the ethyl ether and the addition of petroleum ether is to render less soluble the milk sugar or other non-fatty solids which would be dissolved by ethyl ether alone.

The method, while applicable to whole milk, is especially valuable in determining fat in such products as skim milk or buttermilk which are low in fat. In such cases it is better to use 10 c.c. of milk and double the quantity of reagents.

Milk Sugar.— The sugar in milk is most readily determined by its reducing action on Fehling's solution.

Munson and Walker Method.* — *Directions.* — Measure 25 c.c. of milk into a 500-c.c. graduated flask. Add about 400 c.c. of water, 10 c.c. of copper sulphate solution,† then 35 c.c. of tenth-normal sodium hydroxide (or an equivalent quantity of a stronger solution) and make up to 500 c.c. Mix thoroughly and filter through a dry filter.

In a No. 3 beaker mix 25 c.c. of the Fehling's copper sulphate solution and 25 c.c. of the alkaline tartrate solution. Add 50 c.c. of the milk sugar solution, prepared as above, cover the beaker with a watch glass, and heat it upon wire gauze. Regulate the flame so that boiling shall begin in four minutes, and continue the boiling for *exactly two minutes.*

Filter the cuprous oxide *without delay* through asbestos in a weighed Gooch crucible, wash it with hot water until free from alkali, pour out the hot filtrate, then wash with 10 c.c. of alcohol and, finally, with 10 c.c. of ether. Dry the crucible for 30 minutes at the temperature of boiling water and weigh. Find the milligrams of lactose monohydrate corresponding to the weight of cuprous oxide from Table XII on page 221 and calculate the percentage present in the milk.

Notes. — Before the lactose can be determined by Fehling's solution the protein and fat must first be removed. This is done by the precipitation with copper hydroxide, the fat being carried down mechanically by the precipitated protein. The addition of alkali should be such that a slight excess of copper still remains in solution, since an excess of alkali will prevent the precipitation of part of the protein. The quantity stated in the procedure is correct for most milks.

On account of the considerable dilution of the sample, the volume of the precipitated protein and fat need not be considered.

The general principle upon which all these methods depend

* *J. Am. Chem. Soc.*, *1906*, 663; *1907*, 541.

† 69.28 grams per liter. The copper sulphate solution used in the Fehling determination may be conveniently employed.

is based on the fact that certain sugars, among which is lactose, have the power of reducing an alkaline solution of copper to a lower state of oxidation in which copper is separated as cuprous oxide. The copper salt which is found to give the most delicate and reliable reaction is the tartrate. The two solutions which make up the Fehling's solution are best preserved separately, and mixed only when wanted for use, as otherwise the reducing power of the solution is liable to change.

The amount of reduction of the copper salt to the cuprous oxide is affected by the rate at which the sugar solution is added, the time and degree of heating, and the strength of the sugar solution; hence the necessity for adopting a definite procedure and for taking the results from a table determined by exactly the same procedure for varying amounts of the sugar.

The asbestos which is used should be previously boiled in nitric acid and then in dilute sodium hydroxide and thoroughly washed. A layer about a centimeter thick should be used in the crucible, and a "blank" determination made with the Fehling's solution should not show a change in weight greater than one-half milligram. After the precipitated cuprous oxide has been weighed it may be dissolved in hot dilute nitric acid, and the asbestos in the crucible washed and dried as described, when it is again ready for use. Do not remove the asbestos from the crucible.

Proteins. Determination of Total Protein. — This is best done by the Kjeldahl method. Weigh 5 grams of milk into a Kjeldahl flask, add 10 c.c. of concentrated sulphuric acid and three drops of mercury and carry out the determination as described on page 182.

The tendency of the alkaline solution to froth during the distillation, which is especially noticeable with milk, can be prevented by the addition of a piece of paraffin the size of a pea. Multiply the per cent of nitrogen by the factor 6.38 to obtain the per cent of protein.

Separation of Casein and Albumin. — The usual method of precipitating the casein by acid at a temperature below the

coagulating point of the albumin, while capable of good results, is tedious and rather unsatisfactory except after considerable experience. The following volumetric method, devised by Van Slyke and Bosworth * gives results of almost equal accuracy, but requires much less time and skill.

Measure 20 c.c. of the well-mixed milk into a 200-c.c. graduated flask and add about 80 c.c. of water. Add 1 c.c. of phenolphthalein solution and tenth-normal sodium hydroxide until a faint pink color remains throughout the mixture even after considerable shaking. Avoid an excess of alkali.

To the neutralized diluted sample, which should be at a temperature of 18° C. to 24° C., add tenth-normal acetic acid in 5-c.c. portions, shaking vigorously for a few seconds after each addition. After thus adding 25 c.c. and shaking, the mixture is allowed to come to rest. If enough acid has been added, the casein separates promptly in large, white flakes, and on standing a short time, the supernatant liquid appears clear, not at all milky. If the addition of 25 c.c. of acid is insufficient to separate the casein properly, add 1 c.c. more of acid and shake; continue this addition of acid 1 c.c. at a time, until the casein separates promptly and completely upon standing a short time. Note the number of c.c. of acid used.

After the casein is completely precipitated make up the mixtures to the 200-c.c. mark with water, shake thoroughly and filter through a dry filter. Filtration should be rapid and the the filtrate quite clear. If a marked turbidity is apparent in the filtrate, a new sample should be taken and the process repeated, using more acid than before. Titrate 100 c.c. of the filtrate with tenth-normal sodium hydroxide and phenolphthalein to a pink color which remains throughout the solution for thirty seconds. Subtracting the number of c.c. of sodium hydroxide from one-half the c.c. of tenth-normal acetic acid added will give the c.c. of acid required to precipitate the casein for 10 c.c. of milk. (1 c.c. of $\frac{N}{10}$ acetic acid = 0.11315 gms. of casein.)

* *J. Ind. Eng. Chem.*, *1909*, 768.

Calculation of Milk Solids. — It has long been recognized that in normal milk the constituents are present in a fairly constant ratio. This being true, it should be possible, having determined two factors, to find a third by calculation, or at least to show by such calculation a sufficient variation from the normal to indicate the adulteration of the sample. For example, given the lactometer reading and fat, to calculate the total solids:

L = the lactometer reading,
s = increase in lactometer reading by 1 per cent solids not fat,
f = decrease in lactometer reading by 1 per cent fat,
T = total solids,
S = per cent of solids not fat,
F = per cent of fat.

Then $L = Ss - Ff,$
Since $S = T - F$
$L = (T - F)\, s - Ff,$
whence $T = \dfrac{L + Ff}{s} + F.$

The uncertainty of the calculation lies in the values for s and f, which, on account of the difference in solution densities of the components of the solids not fat, are not absolute constants.

Based on the principle just stated, various formulæ have been proposed for the calculation of milk solids. One of the simplest of these is that of Hehner and Richmond *

$$T = \frac{L}{4} + 1.2\, F + 0.14,$$

where T is the per cent of total solids, L the reading of the lactometer, and F the fat.

When a number of calculations are to be made, Richmond's "Milk Scale" will be found convenient. This is an instrument based on the principle of the slide-rule, having three scales, two of which, for the fat and the total solids, are marked on

* *Analyst, 1888,* 26; *1892,* 170.

the body of the rule, while that for the lactometer readings is marked on the sliding part.

A similar relation has been worked out for the protein, so that if a constant value be assumed for the ash, the composition of a sample may be determined with a fair degree of approximation from the two simple determinations of specific gravity and Babcock test.

The relation between the protein and fat has been expressed by Van Slyke* as $P = 0.4\ (F - 3) + 2.8$. Similarly Olsen † has proposed the following formula for calculating the protein from the total solids (T.S.):

$$P = \text{T.S.} - \frac{\text{T.S.}}{1.34}.$$

These values will naturally be most nearly correct in the case of normal average milk. With watered or skimmed milk they will be only approximate.

In the table below the values calculated for a sample are compared with those actually determined:

Determination.	Actual values.	Calculated values.
Lactometer reading	33	.....
Fat (Babcock)	3.80	.....
Total solids	12.73	12.95
Ash	0.71	0.7 (assumed)
Proteins	3.33	{ 3.12 (Van Slyke) { 3.29 (Olsen)
Milk sugar	5.04	5.16
Solids not fat	8.93	9.15

Examination of Milk Serum. — The most variable constituents of normal milk are the fat and protein, especially the former; the least variable are the ash and milk sugar. The milk serum, or milk from which the fat and protein have been removed, is, therefore, of more uniform composition than the milk itself, hence better suited for the detection of adulteration and espe-

* *J. Am. Chem. Soc.*, *1908*, 1182.

† *J. Ind. Eng. Chem.*, *1909*, 253.

cially of added water. The serum may be prepared by adding to the milk some suitable precipitant of the protein, as calcium chloride, acetic acid or copper sulphate. The clear liquid after filtration may be examined for its content of dissolved solids, its specific gravity or most conveniently by the immersion refractometer.

*The Copper Sulphate Method.** — Dissolve 72.5 grams of crystallized copper sulphate in water and dilute to a liter. This solution should be adjusted, if necessary, so that it will refract at 36 degrees on the scale of the immersion refractometer at 20° C. or have a specific gravity of 1.0443 at 20° C. compared with water at 4° C. To one volume of the copper solution add four volumes of milk, shake well and filter. The filtrate will usually be clear after the first few drops have passed through. On the clear filtrate either the refraction at 20° C., the specific gravity $\left(\frac{20^\circ\text{ C.}}{4^\circ}\right)$ or the total solids may be determined.

Notes.—Examination of the copper serum from 150 samples of known purity milk gave refractions varying from 36.1 to 39.5, while the total solids of the same samples showed a range from 17.17 per cent to 10.40 per cent and the fat varied from 7.7 per cent to 2.45 per cent.

The minimum values for the copper serum of normal milk are 36 degrees for the refraction at 20° C., 1.0245 for the specific gravity $\left(\frac{20^\circ}{4^\circ}\right)$ and 5.28 per cent for total solids.

If the milk is already soured, it may be filtered and similar determinations made on the natural sour serum, which for unwatered milk should not refract below 38.3 or have a specific gravity at $\frac{20^\circ\text{ C.}}{4^\circ}$ below 1.0229.

SPECIAL TESTS FOR ADULTERANTS

Cane Sugar. — Cane sugar may be present in milk from diluted condensed milk used to eke out the supply or may be present from calcium saccharate, added as a thickening agent.

* Lythgoe: *Ann. Rpt. Mass. Bd. Health, 1908,* 594.

It is evident that any considerable amount which had been added could be detected by the taste.

To detect the presence of cane sugar boil about 10 c.c. of the milk with 0.1 gram of resorcin and 1 c.c. of strong hydrochloric acid for five minutes. The liquid will be colored rose-red if cane sugar is present. The color produced by heating should not be confused with the pink color which may appear in the cold if the milk contain certain coal-tar colors.

A similar test is the reduction of ammonium molybdate. As recommended by Cotton * 10 c.c. of the milk are mixed with 0.5 gram of powdered ammonium molybdate and 10 c.c. of dilute (1 to 10) hydrochloric acid are added. In another tube 10 c.c. of milk known to be free from sucrose are similarly treated and the tubes placed in a water-bath, the temperature of which is gradually raised to about 80° C. If sucrose is present, the milk will gradually turn deep blue, while genuine milk remains unchanged unless the temperature approaches the boiling point. Cotton states that the reaction will detect as little as 1 gram of cane sugar in a liter of milk.

Note. — Both of these tests, although used to detect cane sugar, are in reality tests for levulose, formed in this case by the partial inversion of the sucrose.

Preservatives. — The preservatives most commonly employed in milk are formaldehyde, boric acid or borax, and mixtures of the two, and possibly hydrogen peroxide and fluorides. Salicylic acid and sodium benzoate, although largely used in some other classes of food materials, have been reported very rarely as present in milk.

Formaldehyde. — This is the ideal preservative for milk, being readily used and by far the most efficient. Quantities which give a proportion in the milk of from 1 in 10,000 parts to 1 in 50,000 are ordinarily employed. Such an amount will suffice to preserve the milk for from 24 hours to several days. Larger quantities, such as 1 part in 3000, will preserve the milk for months. These large amounts, however, would be more or

* *J. Pharm. Chim., 1897*, 362.

less apparent by the taste or odor. A tabular statement showing the efficiency of formaldehyde in preserving milk as compared with boric acid, borax and sodium carbonate will be found in Leach's Food Analysis.

Several of the best tests for detecting formaldehyde are described below. These may be applied directly to 10 c.c. of the milk, or as suggested in the gallic acid test, a larger quantity, 25 to 100 c.c., may be distilled and the test applied to the first portion of the distillate.

(1) When the sulphuric acid is added to the milk in making the Babcock test for fat, a bluish-violet ring will be noticed at the junction of the two liquids when formaldehyde is present. One part of formaldehyde in 200,000 parts of milk can be detected by this test, but it fails when the formaldehyde amounts to 0.5 per cent. The test is more delicate if the sulphuric acid contains a trace of ferric chloride.

(2) To 10 c.c. of milk in a small porcelain dish add an equal volume of hydrochloric acid (1.20 sp. gr.). Add one drop of ferric chloride solution and heat the dish with a small flame, stirring vigorously, until the contents are nearly boiling. Remove the flame and continue the stirring for two or three minutes, then add about 50 c.c. of water. The presence of formaldehyde will be shown by a violet color which appears in the particles of the precipitated casein, the depth of color depending on the amount of formaldehyde present. The color should be observed carefully at the moment of dilution. This test readily shows the presence of one part of formaldehyde in 250,000 parts of milk, if fresh.

(3) *Gallic Acid Test.** — This test has been found by Sherman† to be much more delicate than either of the preceding tests. 25 to 50 c.c. of the milk should be acidulated with phosphoric acid and distilled. To the first 5 c.c. of the distillate add 0.2 to 0.3 c.c. of a saturated solution of gallic acid in pure ethyl

* Barbier and Jandrier: *Ann. Chim. anal.*, 1, 325; Mulliken and Scudder: *Am. Chem. J.*, *1900*, 444.

† *J. Am. Chem. Soc.*, *1905*, 1499.

alcohol and pour it cautiously down the side of an inclined test tube containing 3–5 c.c. of pure concentrated sulphuric acid. If formaldehyde is present a green zone is formed at the junction of the two layers, gradually changing to a pure blue ring.

The delicacy of the test is about one part of formaldehyde in 500,000 parts of milk.

Notes. — It should be borne in mind that when small amounts of formaldehyde are added to milk the ordinary tests will show the presence of the preservative for only a short time. For example, it has been shown by Williams and Sherman * that when formaldehyde was added to milk in the proportion of 1 part to 100,000 only a faint test was given after 48 hours standing; and that the preservative had entirely disappeared in from three to five days. This is due to the gradual formation of condensation products of the formaldehyde with the proteins of the milk which do not respond to the usual reaction. In such a case, it is better to distill the milk as directed and apply the test with gallic acid to the distillate. The test is thus made more delicate, so that the preservative may still be shown when the simpler tests have failed.

Another possible contingency is that some substance may be added with the formaldehyde which will interfere with the tests for its detection. Both hydrogen peroxide and nitrites prevent the reaction of formaldehyde in the usual tests and preservatives are on the market which are mixtures of formaldehyde with hydrogen peroxide or a nitrite. The sulphuric acid test and the hydrochloric acid-ferric chloride test can be used to show the formaldehyde in the presence of considerably larger quantities of nitrites by first removing the latter. Add to 10 c.c. of the milk 1 c.c. of a 10 per cent solution of urea, then 2 c.c. of dilute (1:40) sulphuric acid and immerse the test tube in boiling water for two minutes. Cool and carry out the test as usual. The reaction between the urea and the nitrous acid may be expressed:

$$CO(NH_2)_2 + 2\,HNO_2 = CO_2 + 2\,N_2 + 3\,H_2O.$$

* *J. Am. Chem. Soc.*, *1905*, 1497.

Boric Acid or Borax. — Make 25 c.c. of the milk distinctly alkaline with lime water and evaporate to dryness on the water-bath. Char the residue over a flame but do not necessarily heat it until white. Digest the residue with 15–20 c.c. of water and add hydrochloric acid (1.12) until the mixture is faintly acid to litmus paper. Filter, and add 1 c.c. of acid in excess. Place a strip of turmeric paper in the solution and evaporate to dryness on the water-bath. If boric acid or borates are present, the paper takes on a peculiar red color, which is changed by ammonia to a dark blue-green, but is restored by acid. Excess of hydrochloric acid should be avoided, as it turns the paper a dirty green when evaporated. This test can also be applied to the hydrochloric acid solution of the ash.

Sodium Carbonate. — Detected in the milk-ash, as on page 141. If effervescence occurs, test the original milk with rosolic acid as follows: Mix 10 c.c. of milk with an equal volume of alcohol, and add a few drops of a one per cent solution of rosolic acid. The presence of sodium carbonate is indicated by a more or less distinct pink coloration. A comparative test should be made at the same time with milk known to be pure.

Salicylic and benzoic acids. — If it is desired to test for these, the following method may be employed. To 25 c.c. of milk add 100 c.c. of water and precipitate the proteins and fat with copper sulphate and sodium hydroxide, as described on page 145. Filter and add to the filtrate 5 c.c. of concentrated hydrochloric acid. Extract with ether and proceed as outlined on page 196.

Coloring Matter. — The object in adding coloring matter to milk is in general to disguise the bluish appearance of skimmed or watered milk. For this reason it is rather unusual to find added color in the case of milk which is of standard quality, although such cases have been reported.

Formerly the chief color used was annatto, a reddish-yellow coloring matter obtained from the seeds of *Bixa Orellana*, a shrub growing in South America and the West Indies. A solution of the color in very dilute alkali is employed. More recently

various coal-tar dyes and even caramel have been used. The latter is, perhaps, not so likely to be found, because its color is too brown and not enough yellow to give the desired creamy appearance to the milk which is so easily obtained with annatto. The coal-tar colors, especially mixtures of yellow and orange azo dyes, give very good results.

Leach * has suggested a general scheme for the identification of these colors in milk, which with some modifications which experience in the writer's laboratory has shown to be helpful in detecting annatto especially, is given below.

Procedure. — Place about 100 c.c. of the milk in a small beaker, add 3–4 c.c. of 25 per cent acetic acid (sp. gr. = 1.04), stir thoroughly and allow the beaker to stand quietly on the water-bath for about ten minutes, the casein being thus separated as a compact cake. Decant off the whey, squeezing the curd as dry as possible with a spatula. Transfer the curd to a flask, cover it with ether, stopper tightly, and shake the flask violently in order to break up the curd as much as possible. Let it stand for several hours, preferably over night.

Pour off the ether, which contains the annatto, and evaporate (*away from a flame*) until no odor of ether remains. Add 5 c.c. of water and then dilute sodium hydroxide until the mixture, after thorough stirring with a glass rod, is faintly alkaline to litmus paper, and filter through a wet filter. If annatto is present it will permeate the filter and give it an orange-brown color which may readily be seen if the filter is removed from the funnel and the fat washed off under the tap. Its presence may be confirmed by touching the colored portion of the paper with a drop of stannous chloride, which gives a pink color with annatto.

After pouring off the ether examine the milk-curd for caramel or coal-tar color. If the curd is left white, neither of these colors is present. If caramel has been used, the curd will be of a pinkish-brown color; if the color is due to the coal-tar dye, the curd will have a yellow or orange tint. If now some concentrated hydrochloric acid is poured over the curd, the color

* *J. Am. Chem. Soc.*, 1900, 207.

will change immediately to a bright pink with the coal-tar colors ordinarily used.

Notes. — When the milk is curdled by the acid, any added color is carried down by the curd. When this is subsequently treated with ether the fat and annatto are dissolved, leaving any caramel or coal-tar color still in the curd. Since the detection of the two latter colors may depend upon recognizing color in the curd, this should always be compared with the curd prepared in the same manner from a sample of milk known to be free from color.

The ordinary tests for caramel as used to show its presence in distilled liquors or vanilla extract are not sufficiently delicate to detect the extremely small quantity which suffices to impart the desired shade of color to the milk. The color imparted to the curd, however, is characteristic and readily recognized.

It is possible that coal-tar dyes may be used which do not give the pink reaction with hydrochloric acid, since this is characteristic in general only of the azo class of dyes. Even in these cases, however, the orange color of the dye is readily perceptible in the separated curd.

Milk colored with an azo dye may occasionally fail to show its presence if the sample is old or partly decomposed before being tested. This has been shown by Blyth * to be due to the reduction of the dye by nascent hydrogen produced by the growth of certain anaërobic organisms.

Interpretation of Results. — Apart from the addition of foreign ingredients, such as colors and preservatives, which are detected by the specific tests described, the most common forms of adulteration are the addition of water and the removal of cream. By reference to the table on page 136, it will be seen that on account of the variation in the composition of unadulterated cow's milk the detection in all cases is not an easy problem. The variation in the fat content, especially, makes it more difficult to show with certainty the partial removal of cream than the addition of water.

* *Analyst, 1902,* 146.

This is well shown in the following table in which "*A*" is a normal milk, "*B*" the same milk in which the fat has been reduced to 3.6 per cent by adding water and "*C*" the same milk in which the fat has been reduced to 3.6 per cent by skimming.

	A	*B*	*C*
Total solids	12.78	11.34	12.09
Fat	4.00	3.60	3.60
Protein	2.89	2.60	2.91
Sugar	5.00	4.50	4.98
Ash	0.71	0.64	0.72
Solids not fat	8.78	7.74	8.61

It is seen that in sample *C* it is only the fat that has been decreased to any degree. In fact there is nothing in the figures given for *C* to indicate in any way that the sample is not genuine milk, while in *B* the solids not fat are so low as to show the adulteration quite plainly.

Composition of Milk of Known Purity. — The average composition of milk, together with the usual and the extreme limits of variation, have already been stated on page 136. The greater number of published analyses of genuine cows' milk have been limited to determination of solids, fat and specific gravity. A more detailed study, including the constants of the copper serum, will be found in the following table,* which includes the analyses of 33 samples of known purity milk from individual cows, and 4 samples of herd milk, arranged in the order of their percentage of total solids.

In collecting the samples milk was taken from the heaviest milkers, so as to include a larger proportion of low-grade milk for minimum values. None of the milk could be called exceptionally high grade, as samples were not collected from Jersey or Guernsey cows.

Inspection of this table shows, as would be expected, a great variation in the percentage of fat in the individual samples, the highest being almost 100 per cent higher than the minimum values. The solids not fat are seen to present a much less

* Lythgoe: *Bull. Mass. Bd. Health, 1910,* 422.

variation, and as Lythgoe has pointed out, this variation is due very largely to the changes in protein content, the milk sugar and ash remaining fairly constant. Upon this fact depends the special value of an examination of the milk serum.

In some cases all that may be necessary is to show by the analysis that the milk does not conform to the legal standard. In certain of the states, however, a legal distinction is made between milk which is simply below standard and milk which has been actually adulterated by skimming or watering. It is therefore of importance to show by the analysis whether water has been added to the milk directly and not through the breed or feed of the cow.

Detection of Watered Milk. — Since in general the water that has been added is no different from the water already present in the milk it is evident that this form of adulteration can be detected only by showing chemical or physical changes in the milk that could be ascribed only to the addition of water. Methods have been proposed, it is true, based on differences in the added water, such as an abnormally high amount of nitrates, which might have been derived from the polluted barnyard well, but these methods are of little importance.

(a) *Solids Not Fat.* — Since the variation in proportion of solids not fat in normal milk is much less than the range of total solids this is of distinct value in showing added water. Although as indicated in the table of limiting values on page 136, the value for solids not fat may go as low as 7.5 per cent, this is rather uncommon, and a fairer minimum would be 7.7 per cent. A value below 7.7 per cent would certainly be suspicious of added water and if accompanied by correspondingly low values for the constants of the serum could be regarded as direct evidence of adulteration.

(b) *Milk Sugar.* — As suggested by Lythgoe,* the milk sugar may be employed to even greater advantage than the solids not fat in showing adulteration. Knowing the percentage of solids

* *Loc. cit.*

Breed.	Time since calving (mos.).	Weight of milk (lbs.).	Specific gravity, 15°.	Total solids (per cent).	Fat (per cent).	Proteins (per cent).	Ash (per cent).	Solids not fat (per cent).	Sugar (per cent).	Copper serum.				Natural sour serum.		
										Refraction, 20°.	Specific gravity, $\frac{20°}{4°}$.	Solids (per cent).	Sugar (per cent).	Refraction, 20°.	Specific gravity, $\frac{20°}{4°}$.	Ash (per cent).
Grade Durham	2	15	1.035	14.58	5.10	3.35	0.81	9.48	5.00	39.7	1.0280	6.28	4.43	44.4	1.0292	0.768
Holstein	2	15	1.034	13.65	4.50	3.33	0.72	9.15	4.95	38.4	1.0271	6.05	4.54	43.0	1.0273	0.776
Grade Durham	2	16	1.036	13.52	4.00	3.54	0.72	9.52	5.20	38.9	1.0272	6.09	4.38	43.0	.....	.....
Grade	1	20	1.033	13.36	4.30	3.25	0.81	9.06	5.00	38.3	1.0270	5.96	4.63	42.0	1.0279	0.816
Grade	4	18	1.034	13.30	4.00	3.54	0.76	9.30	5.00	38.7	1.0269	6.07	4.56	43.1	1.0280	0.830
Holstein	5	10	1.032	13.29	4.40	3.14	0.70	8.89	4.70	38.8	1.0275	6.10	4.34	42.2	1.0265	0.784
Grade Durham	2	15	1.034	13.27	4.00	3.36	0.78	9.00	5.40	39.2	1.0282	6.20	4.50	43.3	.....	.....
Grade Ayrshire	1	12	1.033	13.26	4.20	2.84	0.66	9.06	5.50	39.7	1.0281	6.39	4.78	43.9	1.0286	0.750
Grade Swiss	2	16	1.033	13.20	4.35	3.29	0.76	8.85	4.75	38.6	1.0261	5.86	4.48	40.8	1.0259	0.788
Grade Durham	2	15	1.033	13.06	3.80	3.37	0.69	9.26	5.75	39.6	1.0271	6.05	4.82	42.4	1.0274	0.750
Grade Durham	2	16	1.034	13.02	4.10	3.01	0.80	8.92	5.30	30.3	1.0277	6.19	4.47	43.1	1.0286	0.769
Ayrshire	9	10	1.031	12.91	3.90	3.51	0.76	9.01	4.35	37.3	1.0259	5.73	3.94	41.0	1.0254	0.846
Grade Ayrshire	2	21	1.031	12.85	4.30	3.15	0.68	8.55	5.20	37.1	1.0259	5.66	4.40	39.9	1.0254	0.760
Grade Holstein	2	17	1.032	12.78	4.00	2.89	0.71	8.78	5.05	38.0	1.0274	6.07	4.40	42.1	1.0272	0.768
Grade Holstein	2	18	1.032	12.78	4.00	2.89	0.69	8.78	4.90	38.5	1.0271	6.05	4.40	42.2	1.0269	0.772
Holstein	6	10	1.031	12.66	3.90	2.97	0.76	8.76	4.35	37.6	1.0268	5.85	4.21	41.8	1.0266	0.820
Grade Holstein	2	20	1.032	12.64	3.80	3.01	0.62	8.84	5.15	38.7	1.0273	6.11	4.71	41.9	1.0270	0.744
Grade Durham	2	18	1.035	12.58	3.70	3.12	0.74	8.88	5.00	38.4	1.0269	5.93	.....	41.9	1.0271	0.741
Grade Durham	2	18	1.034	12.54	3.50	2.97	0.68	9.04	5.25	38.8	1.0274	6.06	4.14	42.7	.....	.....
Grade Durham	2	15	1.033	12.50	3.30	3.08	0.74	9.00	5.05	38.6	1.0271	6.03	4.14	42.7	.....	.....
Holstein	4	16	1.031	12.29	3.80	2.65	0.72	8.49	4.85	37.7	1.0266	5.87	4.38	41.8	1.0264	0.755
Holstein	1	10	1.033	12.27	3.40	3.27	0.84	8.87	4.60	37.6	1.0264	5.82	4.11	41.2	1.0259	0.868
Grade Holstein	8	18	1.032	12.12	3.50	3.32	0.74	8.62	4.40	37.5	1.0258	5.75	4.25	41.4	1.0262	0.780
Grade Ayrshire	3	16	1.032	12.08	3.70	2.80	0.78	8.38	4.65	37.0	1.0259	5.70	4.33	40.6	1.0249	0.804
Grade Holstein	4	20	1.032	12.03	3.45	2.89	0.72	8.58	5.00	38.6	1.0273	6.00	4.43	42.1	1.0270	0.800
Grade Holstein	1	20	1.034	12.00	3.10	2.99	0.71	8.90	5.05	38.4	1.0271	6.07	4.61	41.5	1.0270	0.780
Grade Holstein	4	16	1.030	11.77	3.70	2.67	0.71	8.07	4.50	37.2	1.0261	5.79	4.30	39.7	1.0248	0.740
Holstein	½	18	1.033	11.40	3.20	2.48	0.72	8.20	5.00	37.5	1.0261	5.75	4.22	41.0	1.0258	0.800
Holstein	7	15	1.031	11.27	3.15	3.09	0.78	8.12	4.30	36.6	1.0253	5.47	3.73	39.7	1.0244	0.804
Holstein	10	18	1.031	11.21	3.35	2.81	0.75	7.86	4.30	36.3	1.0250	5.37	3.88	38.7	1.0239	0.792
Holstein	3	20	1.031	11.48	3.40	2.78	0.80	8.08	4.50	36.8	1.0256	5.53	4.02	40.6	1.0255	0.800
Grade Holstein	4	16	1.030	10.66	2.85	2.66	0.65	7.81	4.40	36.4	1.0254	5.51	4.22	39.1	1.0242	0.740
Holstein	1	28	1.030	10.20	2.65	2.40	0.65	7.55	4.50	36.5	1.0259	5.45	3.76	38.6	1.0234	0.735
Mixed milk[1]	.....	.....	1.033	13.40	4.20	3.13	0.70	9.20	5.10	38.5	1.0260	6.05	4.09	42.2	1.0272	0.780
Mixed milk[2]	.....	.....	1.033	13.08	4.10	3.23	0.76	8.98	4.65	37.7	1.0262	5.84	4.34	41.3	1.0259	0.788
Mixed milk[3]	.....	.....	1.033	12.73	3.80	3.33	0.71	8.93	4.70	38.0	1.0261	5.94	4.22	41.4	1.0272	0.768
Mixed milk[4]	.....	.....	1.032	12.53	3.70	3.35	0.73	8.83	4.75	37.7	1.0259	5.77	4.28	41.5	1.0261	0.808

[1] Grade Ayrshire, Durham, Shorthorn and Holstein cows.
[2] Grade Jersey, Ayrshire and Holstein cows.
[3] Grade Holstein and Grade Jersey cows.
[4] Holstein, Grade Holstein and Grade Jersey cows.

and of fat, the protein may be calculated by the formulæ given on page 149. Then if 0.7 be assumed as the value for the ash, the milk sugar may be determined by subtracting from the total solids the sum of the other constituents. The expression for the milk sugar would then become

(1) Milk sugar = T.S. $- (F + [0.4\,(F - 3) + 2.8] + 0.7)$.

(2) Milk sugar = T.S. $- \left(F + \left[\text{T.S.} - \frac{\text{T.S.}}{1.34}\right] + 0.7\right)$.

The portion of the formula enclosed in brackets is the calculated protein in each case. In the case of pure milk the formulæ for calculating the protein will give very similar results, but with adulterated milk they will be divergent, the difference increasing with the extent of adulteration. In the case of watered milk the calculated milk sugar will be too low, ordinarily falling below 4.2 per cent, while, as will be shown later, with skimmed milk, the milk sugar will be too high, generally above 4.8 per cent.

(c) *Milk Serum.* — If the preliminary calculation indicates a possibility of the samples being watered an examination of the serum should be made. This may be done preferably by the copper sulphate method, which is described and the minimum values for pure milk stated on page 150. The following table due to Lythgoe shows the effect of systematic watering on the composition of the milk and the constants of the serum in the case of a milk which was above the average in solids not fat and refraction.

COMPOSITION OF A SAMPLE OF MILK SYSTEMATICALLY WATERED

Added water (per cent).	Solids (per cent).	Fat (per cent).	Solids not fat (per cent).	Copper serum.		
				Refraction, 20°.	Specific gravity, $\frac{20°}{4°}$	Solids (per cent).
0	13.18	4.20	8.98	38.5	1.0272	6.09
10	11.86	3.78	8.08	36.4	1.0249	5.57
20	10.54	3.36	7.18	34.4	1.0233	5.05
30	9.23	2.94	6.29	32.4	1.0211	4.56
40	7.91	2.52	5.39	30.6	1.0194	4.10
50	6.59	2.10	4.49	28.6	1.0174	3.54

It is seen that each 5 per cent of added water lowers the refraction by one scale division, hence with average milk, refracting below 38 degrees, 10 per cent of added water could be detected, and with rich milk 15 per cent can usually be found.

Detection of Skimmed Milk. — Watering milk does not in general change the relation of the various constituents to one another, since these are all reduced in the same proportion, but removing the fat does change these ratios. It is immaterial whether the milk is skimmed by the actual removal of some of the fat or whether separator skim milk is added to normal milk. In either case the resulting product will have its fat content largely reduced, while the proteins and sugar suffer but little change. In normal milk, especially in the mixed milk of a herd, the percentage of fat is rarely less than the protein (see table, page 159). In 5500 analyses of American milks compiled by Van Slyke, with a fat content between 3 and 5 per cent, the average amount of fat was 3.92 per cent and the average amount of proteins 3.20 per cent. If such milk be skimmed the fat may be reduced to 1 per cent or even to 0.1 per cent but the protein content will still be approximately the same as before. In the calculation of milk sugar by the formulæ given on page 160, the same effect will be noticed, that is, the skimming will lower the fat or the solids to a greater extent than the protein. Hence the proteins calculated from the fat or total solids will be too low and the calculated milk sugar will be too high. For practical purposes the limit for unskimmed milk may be set at 4.8 per cent, values above this being suspicious of skimmed milk.

In addition to this preliminary test, the milk may be with certainty declared skimmed if the fat falls below 2.2 per cent, the solids not fat remaining above the average value of 8.5 per cent. If the fat is above 2.2 per cent and below 3.5 per cent, the presence of skimmed milk may be confirmed by making a Kjeldahl nitrogen determination on the suspected sample and calculating the proteins by the factor 6.38. If the proteins

exceed the fat, as stated in the preceding paragraph, the sample is skimmed. If, however, the fat is above 3.5 per cent, this procedure will no longer suffice, since the proteins rarely exceed 3.5 per cent. In these few cases, the skimming can be judged only from the high specific gravity, high solids not fat and correspondingly low fat.

Specific Gravity of Milk Solids. — The specific gravity of the milk solids is sometimes used to show skimming. Fleischmann's formula for calculating this is

$$x = \frac{\text{T.S.}}{\text{T.S.} - \dfrac{(100 \times Gr) - 100}{Gr}}$$

when T.S. = the total solids and *Gr* the specific gravity of the milk.

Example. — A sample of milk contains 12.85 per cent of milk solids and has a specific gravity of 1.031. Required the specific gravity of the milk solids.

$$x = \frac{12.85}{12.85 - \dfrac{(100 \times 1.031) - 100}{1.031}} = \frac{12.85}{12.85 - 3.006} = 1.306.$$

The specific gravity of the solids of normal milk varies between 1.25 and 1.34. It is not changed by watering the milk, but is increased by removing the fat or adding skimmed milk. A value above 1.32 is suspicious while a specific gravity of the milk solids above 1.40 is regarded as conclusive evidence of skimming.

BUTTER

General Statements. — Butter consists of the fat of milk, together with a small percentage of water, salt, and curd. The curd is made up principally of the casein of the milk. These various ingredients are present in about the following proportions:

Fat	78.00–90.0 per cent; average, 82 per cent.
Water	5.00–20.0 per cent; average, 12 per cent.
Salt	0.40–15.0 per cent; average, 5 per cent.
Curd	0.11– 5.3 per cent; average, 1 per cent.

The fat consists of a mixture of the glycerides of the fatty acids. The characteristic feature of butter-fat is the extraordinarily high proportion of the glycerides of the soluble and volatile fatty acids when contrasted with other fats.

The following may be taken as the probable composition of normal butter-fat: *

Acid.	Per cent Acid.	Per cent Triglycerides.
Dioxystearic	1.00	1.04
Oleic	32.50	33.95
Stearic	1.83	1.91
Palmitic	38.61	40.51
Myristic	9.89	10.44
Lauric	2.57	2.73
Capric	0.32	0.34
Caprylic	0.49	0.53
Caproic	2.09	2.32
Butyric	5.45	6.23
Total	94.75	100.00

According to this, the proportion of volatile acids in butter (butyric, caproic, caprylic and capric acids) amounts to 8.35 per cent. The amount of volatile acid in lard, for example, is about 0.1 per cent.

The usual examination of butter consists in the examination of the butter-fat, in order to detect the presence of foreign fats. Those commonly used for this purpose are lard, oleomargarine, and sometimes butter substitutes containing cocoanut oil.

The term "oleomargarine" is usually applied to a mixture of refined lard, "oleo oil," which is mainly the olein of beef fat, and cottonseed oil. Ordinarily a small proportion of butter is added and the product is generally churned with milk.

A comparatively recent form of butter substitute which finds extensive use in some sections of the country is " process," or "renovated," butter. The raw material, or "stock," used for the manufacture of this consists of butter which cannot be sold

* Browne: *J. Am. Chem. Soc.*, *1899*, 807.

as butter either because of deterioration through rancidity or molding or because, through carelessness on the part of the makers, it possesses an unattractive appearance or flavor. The chief recruiting-ground for this material is the country grocery store. The fat, separated from the curd by melting and settling, is aerated to remove disagreeable odors and leave it nearly neutral. This is then emulsified with fresh milk which has been inoculated with a bacterial culture, and the whole is chilled, granulated, and churned. The butter is then worked and packed for market in the usual manner. The character of the product has much improved since the early days of the industry, the best grades now approximating the lower grades of creamery butter.

The "aroma" of butter seems to be connected with the decomposition produced by the action of bacteria on the casein and the small amount of milk-sugar that is present, and not with any change in the fats; there is no evidence, however, that any unwholesome effect is produced by the aroma-giving organisms.

The rancidity of butter-fat is generally considered to be due to decomposition and oxidation of the fatty acids, especially the unsaturated ones, the amount of change depending on conditions of light, heat, and exposure to air.

Analysis of Butter. — Apart from the examination of the butter fat to detect the addition of foreign fats, butter itself is often analyzed in order to determine variations in its constituents from the normal, or the addition of deleterious substances.

The Federal standard for butter describes it as "the clean, non-rancid product made by gathering in any manner the fat of fresh or ripened milk or cream into a mass, which also contains a small portion of the other milk constituents, with or without salt, and contains not less than eighty-two and five-tenths (82.5) per cent of milk fat. By acts of Congress approved August 2, 1886, and May 9, 1902, butter may also contain added coloring matter."

The determinations usually made to ascertain whether the

butter is of standard quality are the water, fat, ash, curd, and salt. Of these the first four can be made on the same weighed sample, following in general the methods recommended by the Association of Official Agricultural Chemists.*

The following method is simpler and gives results comparable with the official methods:

Weigh about 2 grams of butter into a platinum Gooch crucible, half-filled with ignited fibrous asbestos, and dry it at 100° C. to constant weight. The loss in weight is the amount of *water*. Then treat the crucible repeatedly with small portions of petroleum ether, using gentle suction, and again dry it to constant weight. The difference between this and the preceding weight will be the amount of *fat*. Now carefully heat the crucible over a small flame or in a muffle until a light grayish ash is obtained. The loss in weight is the amount of *curd*, and the residual increase in weight over that of the crucible and asbestos is the *ash*. If desired, the *salt* may be washed out of the ash and determined by titration with silver nitrate after neutralizing the solution with calcium carbonate.

Notes. — If the sample for analysis is to be taken from a considerable quantity of butter, great care must be taken in sampling, because the butter is usually not homogeneous in composition and cannot be mixed by stirring. The best plan is to take a fairly large sample of 100 to 200 grams or more, melt it at the lowest possible temperature in a jar or wide-mouthed glass-stoppered bottle and mix by violent shaking. Then cool until sufficiently solid to prevent the separation of the fat and water, taking especial care to shake the sample thoroughly during the cooling.

Rapid methods for the determination of water in butter have been devised by Patrick † and Gray ‡ especially for the examination of large numbers of samples.

Good butter should in general contain not less than the amount

* *Bur. of Chem., Bull.* 107 (*Rev.*), p. 123.
† *J. Am. Chem. Soc.*, 1007, 1126.
‡ *Bur. Animal Ind., Circ.* 100.

of fat required by standard, not more than 2.0 per cent of curd, and not over 16 per cent of water.

Salt. — If a direct determination of salt is desired, the following method, although tedious, will give satisfactory results:

Weigh 10 grams of butter in a small beaker, add 30 c.c. of hot water, and when the fat is completely melted transfer the whole to a separatory funnel. Shake the mixture thoroughly, allow the fat to rise to the top, and draw off the water, taking care that none of the fat-globules pass the stopcock. Repeat the operation four times, using 30 c.c. of water each time. Make the washings up to 250 c.c., mix thoroughly, and titrate 25 c.c. in a six-inch porcelain dish, using $\frac{N}{20}$ silver nitrate with potassium chromate as an indicator.

Preservatives. — About 50 grams of butter are mixed with 25 c.c. of chloroform in a separatory funnel, 100 c.c. of dilute (0.1 per cent) sodium carbonate solution added, and the whole mixed, avoiding violent shaking. After the separation of the layers, which may be greatly aided by a suitable centrifuge, the aqueous layer is examined for preservatives, especially for boric, benzoic and salicylic acids, by the methods described on pages 154 and 196.

Colors. — No methods are described for the detection of colors in butter since these, being allowed, do not constitute an adulteration. If it be desired to test for added color in oleomargarine, methods may be found in Allen's Commercial Organic Analysis, 4th Ed., Vol. II, or in Leach's Food Analysis.

Examination of the Fat. — The fat is first separated from the other constituents of the butter so that it may be weighed out for the various tests.

Directions. — Melt a piece of butter, about two cubic inches, in a small beaker placed on top of the water-bath so that the temperature shall not rise above 50° to 60°. After about fifteen minutes the water, salt, and curd will have settled to the bottom. (A better separation may be secured by dividing the melted sample equally between two test-tubes and whirling them for

3 to 4 minutes in a centrifugal machine.) Place a bit of absorbent cotton in a funnel, previously warmed, and decant off the clear fat through the cotton into a second beaker, taking care that none of the water or curd is brought upon the filter. When the filtered fat has cooled to about 40° place a small pipette in the beaker and weigh the whole.

By means of the pipette the desired amount of fat is taken out, the pipette replaced in the beaker, and the whole again weighed. The difference in weight gives the exact amount of fat taken. It is a saving of time, however, if several portions are to be weighed out, to make the weights one after another, so that one weight will suffice for a determination. Weigh out thus: Two portions of 5 grams each into 250-c.c. round-bottomed flasks for the Reichert-Meissl method, one portion of 2.5 to 3 grams into a 500-c.c. beaker for Hehner's process, two portions of about 0.35 to 0.5 gram each into 300-c.c. glass-stoppered bottles for determination of the iodine value. In the case of the larger portions, weigh only to the nearest milligram.

(1) **Reichert-Meissl Number for Volatile Fatty Acids** — *Directions.* — To the fat in the 250-c.c. flasks add 2 c.c. of strong caustic potash (1 : 1) and 10 c.c. of 95 per cent alcohol. Connect the flask with a return-flow condenser and heat on a water-bath so that the alcohol boils vigorously for 25 minutes. At the end of this time, disconnect the flask and evaporate off the alcohol on a boiling water-bath. After the complete removal of the alcohol, add 140 c.c. of recently boiled distilled water which has been cooled to about 50 degrees. The water should be added *slowly*, a few cubic centimeters at a time. Warm the flask on the water-bath until a clear solution of the soap is obtained. Cool the solution to about 60 degrees and add 8 c.c. of sulphuric acid (1 : 4) to set free the fatty acids. Drop two bits of pumice, about the size of a pea, into the flask, close it by a well-fitting cork, which is tied in with twine, and immerse it in boiling water until the fatty acids have melted to an oily layer floating on the top of the liquid. Cool the flask to about 60 degrees, re-

move the cork, and immediately attach the flask to the condenser.

Distill 110 c.c. into a graduated flask in as nearly thirty minutes as possible. Thoroughly mix the distillate, pour the whole of it through a dry filter, and titrate 100 c.c. of the mixed filtrate with $\frac{N}{10}$ sodium hydroxide, using phenolphthalein as an indicator. Multiply the number of cubic centimeters of alkali used by eleven-tenths, and correct the reading also for any weight of fat greater or less than 5 grams.

For example, if 5.3 grams of butter-fat are used, and 100 c.c. of the distillate require 27.4 c.c. of $\frac{N}{10}$ NaOH, 110 c.c. would require $27.4 \times \frac{11}{10} = 30.14$ c.c. Then $5.3 : 30.14 = 5 : x$. $x = 28.4$. x is the Reichert-Meissl number.

Notes. — The Reichert-Meissl number for genuine butter varies from 24 to 34; the average usually taken is 28.8.

Cocoanut oil gives a value of 6–8; other edible fats and oils have a value usually less than 1.

Cocoanut oil is used, to some extent, as a substitute for butter in confections and crackers, in cooking fats, and also in cocoa-butter substitutes. Its presence is indicated by the Reichert-Meissl number taken in connection with the *saponification value*, that is, the number of milligrams of potassium hydroxide required to saponify one gram of the fat. (For a description of the method of determining this see Lewkowitsch: Oils, Fats and Waxes; or Gill: A Short Handbook of Oil Analysis.) The Reichert-Meissl number is higher in butter fat than in cocoanut oil, while the saponification value is lower. In pure butter fat the value of the expression:

Saponification value — (Reichert-Meissl number — 200) varies from 3.4 to 4.1; in pure cocoanut oil, it runs from 47 to 50.7.*

Another method of value in showing the presence of cocoanut oil is the determination of the Polenske number† which repre-

* Juckenack and Pasternack: *Ztschr. Nahr. Genussm.*, **7**, *1904*, 193.

† Polenske: *Ztschr. Nahr. Genussm.*, *1904*, 273.

sents the volatile fatty acids insoluble in water. This value for butter is from 1 to 3; for cocoanut oil, from 16 to 18. Details of the procedure, which it requires some experience to carry out successfully, may be found in the original paper or in Leach's Food Analysis, 3d ed., page 483.

The reactions involved in the Reichert-Meissl method may be simply explained as follows:

When the fat is treated with potash it is decomposed, the glycerine being set free, and the potassium salts of the fatty acids, that is to say, the potassium soaps, are formed. Hence the process is called *saponification*. For butyric acid the reaction may be expressed,

$$C_3H_5(C_3H_7COO)_3 + 3\ KOH = 3\ C_3H_7COOK + C_3H_5(OH)_3.$$

The alcohol is used to dissolve the fat. But at the moment the butyric acid is set free it tends to combine with the alcohol to form a volatile ether:

$$C_3H_7COOH + C_2H_5OH = C_3H_7COOC_2H_5 + H_2O.$$

The object of the return-flow condenser is to prevent the escape of this volatile ether and to allow of its complete saponification.

If the water used to dissolve the soap is added too rapidly, the soap may be decomposed with the liberation of the fatty acids: $C_3H_7COOK + H_2O = C_3H_7COOH + KOH$.

The fatty acids are set free at the proper time by means of sulphuric acid, and the volatile acids distilled off and titrated. The pumice is added to prevent explosive boiling.

The whole of the volatile acids do not pass over into the distillate, but only a part, the amount depending upon the rate of distillation and the volume of the distillate. Hence, in order to get uniform results, it is necessary to follow the prescribed procedure with great care.

In Great Britain all determinations of the Reichert-Meissl number, which are likely to lead to prosecutions under the Margarine Act, must be made in a specified apparatus, the dimensions of which are definitely stated and the procedure exactly defined.*

* *Analyst*, **25**, *1900*, 309.

Some of the errors in the Reichert-Meissl method may be avoided, and the process materially shortened by carrying out the saponification with glycerol and caustic soda as recommended by Leffman and Beam.* The method is as follows:

Weigh 5 grams of the fat into a 250-c.c. round-bottomed flask and add 20 c.c. of glycerol-soda solution.† Hold the flask with tongs, and heat it directly over a flame until foaming ceases and the mixture becomes perfectly clear, which ordinarily requires about five minutes. Add to the clear soap solution 135 c.c. of water, adding it at first in very small portions to prevent foaming. Finally add the pumice and sulphuric acid, as in the Reichert-Meissl method, and distill without previous melting of the fatty acids.

(2) **Hehner's Method for Direct Determination of the Fixed Fatty Acids.** — *Directions.* — To the portion of 2.5 grams weighed out into the 500-c.c. beaker add 1 c.c. of caustic potash and 20 c.c. of 95 per cent alcohol. Cover the beaker with a watch-glass and heat it on the water-bath until the liquid is clear and homogeneous. As it is not essential to prevent the escape of the volatile acids, the use of a return-flow condenser is not necessary. Evaporate off the alcohol on the water-bath and dissolve the soap in about 400 c.c. of warm distilled water. When the soap is completely dissolved, add 10 c.c. of hydrochloric acid (sp. gr. 1.12), and heat the beaker in the water-bath almost to boiling until the clear oil floats. Meanwhile, dry and weigh a thick filter in a small covered beaker. Allow the solution to cool until the fat forms a solid cake on top; filter the clear liquid and finally bring the solid fats upon the weighed filter. Wash the beaker and fat thoroughly with cold water, then wash out the fat adhering to the beaker with boiling water, which is poured through the filter, taking care that the filter is never more than two-thirds full. If the filter paper is of good texture and thoroughly wet beforehand, it will retain the fatty acids completely. If, however, oily particles are noticed in the

* *Analyst, 1891*, 153.

† 20 c.c. of 50 per cent caustic soda solution to 180 c.c. of glycerol.

filtrate, cool it by adding pieces of ice, remove the solidified particles with a glass rod and transfer them to the filter. Cool the funnel by plunging it into cold water, remove the filter, place it in the weighing-beaker, and dry it at 100° to constant weight. The fat should be heated about an hour at first, then for periods of about thirty minutes, until the weight is constant within 2 mgs.

Notes. — 87.5 per cent is usually taken as the proportion of fixed fatty acids in butter-fat; 88 and 89 per cent have been frequently found. All other fats yield from 95 to 96 per cent of insoluble fatty acids.

(3) **Determination of Iodine Value.** — This method is based on the fact that certain of the fatty acids, notably the "unsaturated acids," as oleic acid, $C_{17}H_{33}COOH$, take up the halogens with the formation of addition products.

Directions. — Dissolve the fat in the 300-c.c. bottles in 10 c.c. of chloroform. Add 30 c.c. of the iodine solution from a pipette or glass-stoppered burette, and allow the bottles to stand with occasional shaking for thirty minutes. Add 10 c.c. of 20 per cent potassium iodide solution and mix thoroughly, then 100 c.c. of distilled water, and titrate the excess of iodine with $\frac{N}{10}$ sodium thiosulphate until the solution is faintly yellow. Add 2 to 3 c.c. of starch solution and titrate to the disappearance of the blue color. Toward the end of the titration shake the bottle vigorously so that any iodine remaining in the chloroform may react with the thiosulphate. Calculate the result in grams of iodine absorbed by 100 grams of fat. This is called the Iodine Number, or Iodine Value.

At the time of making the determination carry out two "blanks" in exactly the same way except that no fat is used.

Standardization of the Thiosulphate Solution. — As this is not permanent, its strength should be determined by means of the standard potassium bichromate solution, 1 c.c. of which is equivalent to 0.01 gram of iodine.

Measure 20 c.c. of the potassium bichromate from a pipette

into an Erlenmeyer flask. Add 5 c.c. of potassium iodide, 100 c.c. of water, and 5 c.c. of strong hydrochloric acid. Titrate the liberated iodine with the thiosulphate solution until the color has almost disappeared, then add starch solution and continue the titration until the blue color disappears, leaving the sea-green color of the chromium chloride. The iodine is liberated in accordance with the following equation:

$$K_2Cr_2O_7 + 14\,HCl + 6\,KI = 8\,KCl + 2\,CrCl_3 + 7\,H_2O + 6\,I.$$

Calculation of Results. — Example. — From the standardization,

16.07 c.c. thiosulphate = 20 c.c. bichromate = 0.200 gram I;
1 c.c. thiosulphate = 0.0125 gram I.

Also, from blank,

30 c.c. iodine solution = 63.60 c.c. thiosulphate.

If 44.85 c.c. thiosulphate were used to titrate the excess of free iodine, 63.60 − 44.85 = 18.75 c.c. is the amount of thiosulphate equivalent to the iodine combined with the fat. If 0.3271 gram of fat were used, since 1 c.c. thiosulphate is equivalent to 0.0125 gram free iodine, $\frac{18.75 \times 0.0125}{0.3271} \times 100 = 71.66$ grams of iodine combined with 100 grams fat.

Notes. — The Iodine Number of butter fat varies between 26 and 38; of oleomargarine, between 60 and 75; of lard, between 46 and 70; of cottonseed oil, from 106 to 110; and of cocoanut oil, between 8 and 9.5.

The products formed by the action of iodine on the fats are mainly addition products with a slight proportion of substituted bodies. Thus the unsaturated *olein*,

$$(C_{17}H_{33}COO)_3C_3H_5,$$

takes up six atoms of iodine, forming an addition product, *di-iodo-stearin*, $(C_{17}H_{33}I_2COO)_3C_3H_5$.

The method in general use for determining the iodine value of fats and oils has been that of Baron Hübl,* an alcoholic solu-

* *Ding. Poly. J.*, 253, 281; *J. Soc. Chem. Ind.*, 3, *1884*, 641.

tion of iodine and mercuric chloride being used as the reagent. The method here described, due to Hanus,* has the advantage that the solutions keep better, remaining practically unchanged for several months, and that the action is about sixteen times as rapid. For the fats and for oils with low iodine values, the results are very close to the figures obtained by the Hübl process. If it is desired to carry out the determination by the older method, directions can be found in any standard work on the analysis of oils.

It should be noted that the "iodine solution" is a solution of iodine bromide in glacial acetic acid, hence great care should be taken that there is no change in temperature between the time of measuring the solution of iodine for the blanks and for the determinations, since the high coefficient of expansion of acetic acid may cause a material error.

Further, the amount of fat taken for the analysis should be such that only a portion of the iodine is absorbed, 60 to 70 per cent being in excess. Care should also be taken to avoid vigorous shaking of the glass-stoppered bottles until near the end of the titration to prevent loss of iodine from the stopper.

(4) **Refractive Index.** — The determination of the refractive index is especially valuable in the examination of butter, and for that matter, in food analysis in general, owing to the rapidity with which the test can be made and the fact that so little of the substance is required. Various forms of refractometers are used for the purpose, a fairly complete description of which will be found in some of the larger works, such as Leach: Food Inspection and Analysis; or Vaubel: Quantitative Bestimmung organischer Verbindungen. The instrument having the widest range is the Abbé refractometer, in which the index of refraction is determined by measuring the total reflection produced by a very thin layer of the melted fat, placed between two prisms of flint glass. This instrument, fitted with water-jacketed prisms, is shown in Fig. 13.

Directions. — Revolve the whole instrument on the axis *b* until

* *Ztschr. Unters. Nahr. u. Genussm.*, 4, 1901, 913.

it reaches the stop provided, then open the prism casing AB by giving the pin v a half-turn (to the right). Be sure that the prism surfaces are clean. If not, clean them carefully with a soft cloth and a little alcohol. Place a few drops of the melted

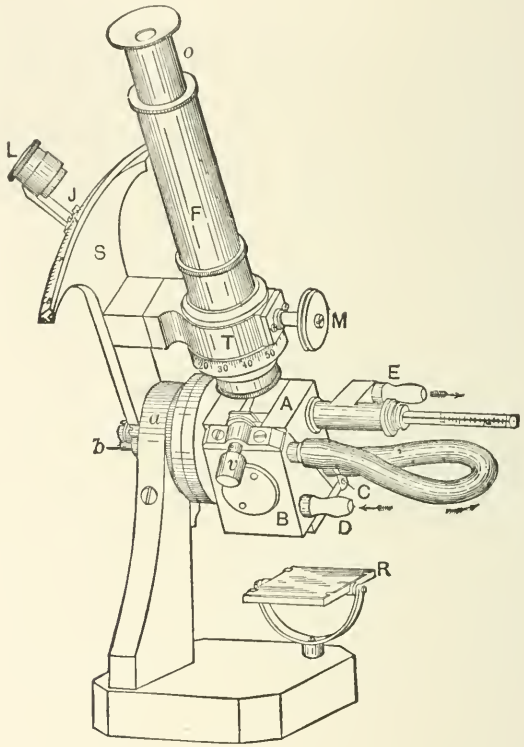


FIG. 13.

sample directly on the surface of the prism and clamp the two together again by turning the pin v in the opposite direction. Now turn the instrument back (toward the observer) as far as possible and bring the "critical line" into the field of vision of the telescope. This is done by holding the sector S firmly with the hand and revolving the double prism by means of the alidade J until the field is divided into a light and a dark portion. If

the line is not sharp focus the ocular of the telescope. If it is colored it is due to dispersion of the light by the liquid and should be corrected by revolving the compensator T by the milled screw M. The correction is made by a system of two revolving Amici prisms in the lower part of the telescope. Adjust the critical line so that it falls on the intersection of the cross hairs of the telescope. Observe the temperature by the thermometer inserted in the prism casing. In the case of solid fats, a sufficiently high temperature should be maintained by a current of warm water to keep the sample well above its melting point. A temperature of 30 to 40° C. is usually sufficient. Do not let the temperature rise above 70° or the prisms may be injured. Read the index of refraction directly through the small lens L, estimating the fourth decimal. Calculate the value for the refractive index at 25° C.

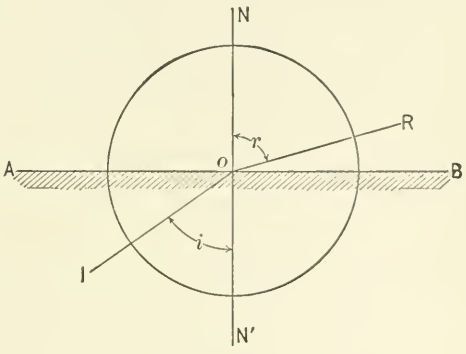


FIG. 14.

Notes. — The principle on which the Abbé refractometer is based will, perhaps, be more clearly understood by reference to Fig. 14.

Let AB be the surface of separation between two media, of which the upper is the rarer, and let a beam of light pass through in the direction IO. It will be seen that as the light passes from the denser to the rarer medium, the angle of refraction r will be greater than the angle of incidence i. If the angle of incidence be increased, then for a certain incident angle, the angle of refraction will become 90°, that is, the refracted ray will coincide with the dividing surface. For incident rays striking the surface at a greater angle than this, the light will be *totally reflected* and there will be no refracted ray. The angle of incidence at

which this occurs is known as the *critical angle.*

Then since $n = \frac{\sin i}{\sin r}$,

at the critical angle $n = \frac{\sin i}{\sin 90^\circ} = \frac{\sin i}{1} = \sin i.$

That is, in passing from a denser to a rarer medium, the index of refraction is equal to the sine of the angle of incidence for the border line of total reflection.

In the Abbé refractometer the refractive index of the liquid is determined by measuring the critical angle for light passing into it from a glass prism of higher refractive index. The sine of this angle is the index of refraction of the liquid, referred to glass, and this multiplied by the refractive index of the glass gives the index of refraction of the liquid referred to air. The divisions on the scale are proportional to the sines of the angles of incidence for total reflection, multiplied by 1.75, the refractive index of the prism and, therefore, give directly the refractive index of the substance examined. Since the light must pass from the denser to the rarer medium, it is evident that the instrument is limited to liquids whose refractive indices are less than 1.75.

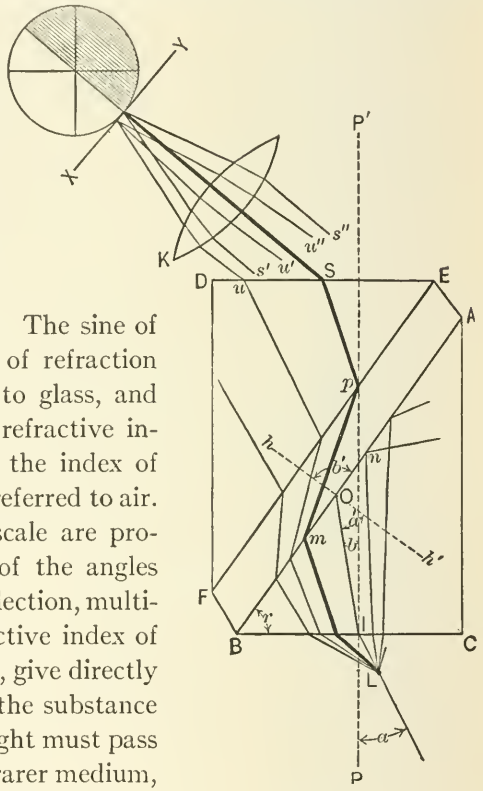


Fig. 15.

Fig. 15, from Browne's Handbook of Sugar Analysis, illustrates diagrammatically the passage of light through the instrument. The heavy line represents the border line of total reflection, the light striking the surface AB at a less angle being

refracted, and illuminating the field of the telescope. The rays which fall upon the surface at a greater angle are totally reflected, leaving the corresponding portion of the telescopic field dark.

The index of refraction decreases with rising temperature. With the common oils and fats, the change for each degree is very nearly a constant, amounting to 0.000365. Leach and Lythgoe * have devised a sliding scale by means of which the temperature correction may be readily made without reference to tables.

The values of n_D^{25} for genuine butter lie between 1.4590 and 1.4620; for oleomargarine the values range from 1.4650 to 1.4700.

The correctness of the instrument should be tested by the "test-plate" which comes with it, cementing it to the prism with monobromnaphthalene, or the testing may be done more conveniently with distilled water. The refractive index of water at ordinary temperatures is given below:

Temperature, °C.	Refractive Index.	Temperature, °C.	Refractive Index.
18	1.3332	23	1.3327
19	1.3331	24	1.3326
20	1.3330	25	1.3325
21	1.3329	26	1.3324
22	1.3328	27	1.3323

Special Tests for Distinguishing Renovated Butter. — *Spoon Test or "Foam" Test.* — Melt a piece of the sample as large as a small chestnut in an ordinary tablespoon or a small tin dish. Use a small flame and stir the melting fat with a splinter of wood (such as a match). Then increase the heat so that the fat shall boil briskly, and stir *thoroughly*, not neglecting the outer edges, several times during the boiling.

Oleomargarine and renovated butter boil noisily, usually sputtering like a mixture of grease and water when boiled, and

* *J. Am. Chem. Soc.*, *1904*, 1193.

produce little or no foam. Genuine butter usually boils with much less noise and produces an abundance of foam, often rising over the sides of the dish or spoon when the latter is removed temporarily from the flame. The difference in regard to the foam is very marked.

Note also the appearance of the particles of curd after the boiling. With genuine butter these will be very small and finely divided, hardly noticeable in fact, while with oleomargarine and renovated butter the curd gathers in much larger masses or lumps.

Notes. — This simple method is of value for giving a quick decision regarding a sample, and is especially useful for the detection of renovated butter. The differences in the composition of butter-fat brought about by renovation are so slight that chemical methods are here of no avail.

The spoon test, however, will distinguish in the great majority of cases between genuine butter on the one hand and oleomargarine and renovated butter on the other; the index of refraction or the chemical methods just described readily distinguish between the two latter.

In genuine butter the curd is somewhat different in composition from that of renovated butter or oleomargarine in that it consists largely of the milk proteins that are insoluble in water, and hence accompany the separated cream. The curd of renovated butter or oleomargarine, on the other hand, comes from the proteins of the milk added directly in the process of manufacture, and consists mainly of coagulated casein. Hence its different appearance in the test.

The crackling and sputtering of the fat in the case of oleomargarine and renovated butter are due to the fact that in the process of manufacture of these the melted fat is sprayed into ice-water, and the cooled particles enclose some water.

Microscopic Examination. — Pure, fresh butter is not ordinarily crystalline in structure. Butter which has been melted, however, and fats which have been liquefied and allowed to cool slowly show a distinct crystalline structure, especially by polar-

ized light. If only fresh butter were sold, and all adulterants had been previously melted and slowly cooled, this method would be all that would be necessary for the detection of adulteration. As it is, however, it is most useful in making comparative examinations of samples which have been subjected to the same conditions.

About the most that can be said is that if a small bit, about the size of a pin-head, of the fresh, unmelted sample, is taken from the center of the mass and pressed out on a slide by gentle pressure on the cover glass, it ought to show a fairly uniform field if examined with a one-sixth objective, using polarized light and a selenite plate. Other fats melted and cooled, and mixed with butter, generally show a crystalline structure and a variegated color with the selenite plate.

In the case of renovated butter, however, there is a distinct difference to be noted in the appearance of the field. With genuine butter the field is much more clear and free from opaque masses of curd than with renovated butter. When the slide is examined by reflected light, turning the mirror so as not to pass light through the slide, these opaque masses in the case of renovated butter show strikingly as white masses against a dark background.

CEREALS

The great importance of cereal food in the diet may be gathered from the fact that dietary studies among a large number of American families have shown that about three-fourths of the vegetable protein, one-half of the carbohydrates, and seven-eighths of the vegetable fat are supplied by the cereals. The reason for such an extensive use of the cereals lies in the fact that, besides being cheap and easily grown, they contain unusually large proportions of nutriment with a very small proportion of refuse. They are readily prepared for the table, are palatable and digestible. In distinction from the two classes of food materials already considered, they are in a dry form, and not liable to rapid change by micro-organisms.

Prepared breakfast foods may be taken as typical and interesting cereal products, and since many of these are somewhat modified from their original composition by cooking or by treatment with malt, the form in which the carbohydrates are present is of almost equal importance with the determination of nitrogen.

The fact that in the breakfast cereals the process of manufacture has in no way increased their actual food value over the grains from which they were prepared, as pointed out in Chapter VIII, is emphasized by the figures in the accompanying table in which some of the most widely-used preparations are compared with the original grains. It will be observed that practically the only change is in the solubility of the carbohydrates, the starch being changed in part to dextrin. In the case of the malted food, the change may go even farther, and a greater or less amount of reducing sugar, principally maltose, be formed.

Moisture. — *Directions.* — Spread about 2 grams of the finely ground material in a thin layer on a watch-glass and dry it in the oven at 100° C. for five hours. On account of the ready absorption of moisture by the dried sample, the use of clipped watch-glasses will be found advantageous.

Note. — With some substances drying in a current of hydrogen or some inert gas may be necessary, but for most cereals the method given will be found satisfactory.

Ash. — *Directions.* — Weigh about 2 grams into a platinum dish, such as is used for the determination of solids in milk, and char it carefully. Ignite at a very low red heat until the ash is white, preferably in a muffle.

Notes. — If a white ash cannot be obtained in this manner, exhaust the charred mass with water, collect the insoluble residue on a filter, burn it, add this ash to the residue from the evaporation of the aqueous extract and heat the whole at a low red heat until the ash is white.

Some cereals, such as whole wheat and barley, will act destructively on platinum dishes, on account of the phosphates present, but can be ignited safely in platinum in the muffle.

COMPOSITION OF CEREAL PRODUCTS.

Name.	Water, per cent.	Protein, per cent.	Fat, per cent.	Crude fiber, per cent.	Ash, per cent.	Soluble in water, per cent.	Reducing sugars, per cent.	Dextrin, per cent.	Starch, per cent.
Wheat	10.5	11.9	2.1	1.8	1.8	.....	.....	.....	71.9
Oats	11.0	11.8	5.0	9.5	3.0	.....	.....	.....	59.7
Corn	10.9	10.5	5.4	2.1	1.5	.....	.....	.....	69.6
Rye	11.6	10.6	1.7	1.7	1.9	.....	.....	.....	72.5
Rice	12.3	6.9	0.3	0.9	0.5	.....	.....	.....	80.0
Barley	10.9	12.4	1.8	2.7	2.4	.....	.....	.....	69.8
Grape Nuts	8.0	12.73	1.57	2.02	1.90	43.76	16.48	14.76	33.15
Force	10.44	11.32	1.50	1.82	3.05	55.15	5.71	14.42	20.20
Toasted Corn Flakes	9.63	9.21	0.54	0.57	1.74	10.34	trace	1.18	70.00
Flaked Rice	11.65	8.78	0.40	1.22	0.29	10.10	trace	trace	68.60
Ralston Wheat Food	11.07	12.55	1.72	1.35	1.20	10.00	trace	4.36	72.86
Cream of Wheat	11.62	13.08	0.87	0.52	0.53	16.78	trace	1.60	67.44
Quaker Oats	9.40	17.55	7.20	2.40	1.89	7.52	trace	5.46	56.06
Shredded Wheat	8.91	11.32	0.87	3.40	1.57	17.90	trace	6.56	57.69

Fat: Ether Extract. — *Directions.* — Place the residue from the determination of moisture, as described above, in a porous paper cup and extract it with pure anhydrous ether for sixteen hours, using the Soxhlet extractor and electric heater as described on page 141. Evaporate off the ether and dry the residual fat at the temperature of boiling water to constant weight.

Note. — The ether extract of cereals is not pure fat but may contain more or less coloring matter or resins. Petroleum ether can be used for the extraction, giving results not essentially different from those obtained with anhydrous ethyl ether.

Total Protein: Determination of Nitrogen by the Kjeldahl Process.* — This method is based upon the decomposition of the nitrogenous material by boiling with strong sulphuric acid. The carbon and hydrogen are oxidized to carbon dioxide and water, a portion of the sulphuric acid being reduced to sulphur dioxide. The nitrogen is left as ammonium sulphate from which the ammonia is liberated by potash or soda and distilled into a known excess of standard acid. The time of digestion can be materially shortened by the use of substances like mercury or potassium sulphate which assist the oxidation or raise the boiling-point of the acid.

Directions. — Transfer about 0.5 gram of the finely divided substance from a weighing-tube to a pear-shaped digestion flask, add 10 c.c. of concentrated sulphuric acid free from nitrogen, and 0.2 gram (three small drops) of metallic mercury. Place a small funnel in the neck of the flask, which should be supported in an inclined position on wire gauze and heated with a small flame until frothing has ceased and the liquid boils quietly. Then increase the heat and boil the solution for at least an hour after it becomes colorless. Allow the flask to cool for a minute or two, and add a few crystals of potassium permanganate until the liquid has acquired a slight green or purple color.

Measure 25 c.c. of $\frac{N}{10}$ acid from a burette into a 300-c.c.

* *Ztschr. anal. Chem.*, **22**, *1883*, 366.

Erlenmeyer flask and place the condenser-tip beneath the surface of the liquid, adding a little water, if necessary, to seal it.

Transfer the digestate with several small portions of distilled water to the distilling flask of the apparatus, add 20 c.c. of potassium sulphide solution, and connect the flask with the condenser. Add 50 c.c. of caustic potash through the separatory funnel, and distill off the ammonia by steam. When 200 c.c. have distilled over, remove the collecting-flask, after rinsing off the condenser-tip with distilled water, and titrate the excess of acid with $\frac{N}{10}$ sodium hydroxide, using methyl orange or cochineal as indicator. If using new reagents, a blank determination should be made with 0.5 gram of cane-sugar in order to reduce any nitrates present which might otherwise escape detection.

Notes. — The temperature during the digestion must be maintained at or near the boiling-point of the acid, since at a lower temperature the formation of ammonia is incomplete.

In some cases, the potassium permanganate is necessary to insure the complete conversion of the nitrogenous bodies into ammonia, although it is probable that its use is unnecessary in the majority of analyses.

The addition of potassium sulphide before distilling is to precipitate the mercury and thus prevent the formation of non-volatile mercur-ammonium compounds.

The Kjeldahl process in the form outlined above is not applicable to the determination of nitrogen in the form of nitrates. In order to render it of more general application various modifications of the method have been proposed, the one generally used in this country being that suggested by Scovell.* In this method salicylic acid is used with the sulphuric acid, being converted by the nitrate into nitro-phenol. By the use of sodium thiosulphate or zinc-dust this is reduced to amido-phenol. The amido-phenol is transformed into ammonium sulphate by the

* U. S. Dept. Agr., *Bull.* 16, *1887*, 51.

heating with sulphuric acid, the use of mercury being absolutely necessary in this case to secure the complete transformation. It is true also that certain other nitrogenous bodies, notably the alkaloids and certain organic bases, do not yield all their nitrogen to the Kjeldahl process without modifications which complicate the method. For a discussion of the efficiency of these various modifications the student is referred to a paper by Sherman and Falk.* In the case of cereals, however, and with the majority of food products, the simpler method outlined will prove entirely satisfactory.

The per cent of proteids may be found by multiplying the per cent of nitrogen by an appropriate factor, the one in general use being 6.25. Recent work has shown, however, that most of the proteids of cereals contain more than 16 per cent of nitrogen, so that the factor 6.25 gives results that are too high. Because all the older work was calculated on this factor, it is still generally used, nevertheless.

Kjeldahl-Gunning Method. — The Gunning method can be used in all cases where the Kjeldahl-Wilfarth modification, just described, is employed, and in some ways it is simpler.

The digestion and distillation are carried out as described on page 182, using the same amount of sample, together with 20 c.c. of concentrated sulphuric acid and 10 grams of powdered potassium sulphate. No mercury and, consequently, no potassium sulphide is used. 100 c.c. of the potash should be added instead of 50.

Note. — The potassium sulphate is added to raise the boiling point of the sulphuric acid and thus shorten the time required for the digestion.

Carbohydrates. — The *total carbohydrates*, often stated in analyses as "nitrogen-free extract," may be readily obtained by subtracting from 100 the sum of the percentages of the other constituents, viz., moisture, ash, ether extract, and nitrogenous bodies. In many cases, however, especially with the cooked or treated cereals and with such classes of cereal preparations as

* *J. Am. Chem. Soc.*, *1904*, 1469.

infant or invalid foods, a further study of the carbohydrates is desirable. These are made up of two general classes: (*a*) *soluble carbohydrates*, including sugars, as sucrose, dextrose and maltose, dextrin and soluble starch, by the latter term being meant starch which is soluble in water but still gives the characteristic blue color with iodine, in distinction from some of the more completely broken-down forms like dextrin, which no longer give blue or purple colors with iodine; (*b*) *insoluble carbohydrates*, including starch, pentosans, lignin bodies, and cellulose. The three latter occur chiefly in the husk or envelope of the grain or in the woody fiber of the plant. The pentosans or gums are distinguished from one another by the formation of specific sugars upon hydrolysis with acids. For ordinary analytical purposes it is sufficient to determine the lignin and cellulose together as "crude fiber." Since the exact procedure to be followed in the determination of the carbohydrates varies largely with each specific case, only a general outline can be presented here.

Sugars. — The finely ground material, previously dried and extracted with ether for the removal of crude fat, is extracted with 85 per cent alcohol. In the extract the reducing sugars may be determined by means of Fehling's solution as described on page 145, and the sucrose determined in the same way after inversion with hydrochloric acid.

Dextrin and Soluble Starch. — The residue from the extraction of the sugars is treated for eighteen to twenty-four hours with water at laboratory temperature with frequent agitation, made up to definite volume, and filtered. This may be tested with iodine, and if no blue color is produced, evaporated to small volume, and the dextrin converted to dextrose by dilute hydrochloric acid and determined by Fehling's solution. In some few cases, however, a blue color with iodine may indicate the presence of soluble starch, in which case an aliquot part of the filtrate may be treated with an excess of barium hydroxide to precipitate the starch. In the filtrate from this precipitate the dextrin is determined by inversion and copper reduction as before. The

difference between the dextrin thus found and the first determination gives the soluble starch.

Starch. — This may be determined in the residue insoluble in cold water by digesting it with malt extract, and determining the dextrose after hydrolysis with dilute acid. It is more common, however, to determine the starch and other insoluble carbohydrates directly on the original material. The methods for the determination of starch vary with the condition in which the starch is found. In the case of nearly pure starch it may be converted into dextrose by boiling with dilute acid, the dextrose being then determined by Fehling's solution in the usual way. Hot acids, however, cannot be used to convert starch in the natural state, as it is found in cereals, because other carbohydrate bodies, especially the pentosans, become soluble under these conditions and the results are too high. In such cases, the starch is brought into solution by treatment with diastase or by heating with water under pressure. The results obtained by direct acid hydrolysis, however, in cases where the highest accuracy is not required, may be sufficient and the method is much quicker and easier of execution than the digestion with diastase.

Direct Acid Hydrolysis. — *Directions.* — Weigh out from 2 to 5 grams of the sample, depending upon the amount of starch present, and wash on a filter with five successive portions of 10 c.c. each of ether. Allow the ether to evaporate from the residue and then wash it with 10 per cent alcohol until free from soluble carbohydrates. 150 c.c. of the dilute alcohol is generally sufficient, but if much reducing sugar or dextrin is present, as may be the case with malted cereals, more will be necessary. Wash the residue from the filter with 200 c.c. of water into a 500 c.c. graduated flask, add 20 c.c. of hydrochloric acid, sp. gr. 1.125, place a funnel in the neck of the flask to retard evaporation, and heat in a boiling water-bath for two and one-half hours. Cool, nearly neutralize with sodium hydroxide and make up to 500 c.c. Filter, and determine dextrose in an aliquot portion, 25 or 50 c.c. of the filtrate, using the method de-

scribed on page 145. Convert dextrose to starch by the factor 0.9.

Note. — The washing to remove soluble carbohydrates is performed with dilute alcohol rather than with water, because the former is less likely to carry starch granules through the paper. The sugar solution when added to the Fehling's solution should be clear and only faintly acid. It should, in general, contain not more than 0.5 per cent of reducing sugar.

Determination with Diastase. — *Directions.* — Treat 2 to 5 grams of the sample with ether and dilute alcohol, as in the previous method, and wash the residue into a 250-c.c. flask with 50 c.c. of water. Heat slowly to boiling, or immerse the flask in boiling water, until the starch gelatinizes, stirring constantly to prevent the formation of lumps. Cool to 55° C., add 20 to 40 c.c. of malt extract, and keep the solution within two degrees of the stated temperature for an hour or until the solution no longer gives the starch reaction with iodine under the microscope. In either case, heat the solution again to boiling to gelatinize any remaining starch granules, test again and if starch is found, cool to 55° C., and treat as before, using a fresh portion of malt extract. Continue this treatment until, when carefully examined under the microscope, a drop of the solution fails to give the iodine reaction for starch. Cool, make up to 250 c.c. and filter through a dry filter. Transfer 200 c.c. of the filtrate to a 500-c.c. graduated flask, add 20 c.c. of hydrochloric acid, sp. gr. 1.125, and carry out the determination as described in the preceding method.

A blank determination must be carried through, using 50 c.c. of water and exactly the same amount of malt extract as used in the regular procedure, in order to correct for the cupric reducing power of the malt extract.

Malt Extract. — Treat 40 grams of fresh coarsely ground malt several hours with 200 c.c. of water, shaking occasionally. Filter and add a few drops of chloroform to prevent the growth of molds.

Notes. — The action of the diastase on the gelatinized starch

is to convert it into maltose and dextrin, that is, into soluble bodies that can be separated by filtration from the pentosans and other carbohydrates that give the high results in the direct acid method. By the action of acid (hydrolysis) the maltose and dextrin are converted to dextrose.

The determination should, if possible, be carried through without interruption. In case this cannot be done, salicylic acid may be used to prevent fermentation, not adding it, however, until after the digestion with diastase.

If the malt itself is not readily procurable, certain forms of prepared diastase are on the market and may be found more convenient either for analytical use or for purposes of illustration. When possible, however, it is preferable to use the freshly prepared malt extract, as the prepared diastase, made at different times and from separate portions of malt, may show great differences in hydrolytic power.

It is sometimes convenient to use freshly collected saliva, this being free from carbohydrate. In this case, the digestion should be carried out at 38° C. instead of 55° C.

Crude Fibre. — The Weende method, the one adopted by the Association of Official Agricultural Chemists, is based on the assumption that the starch and other digestible carbohydrates and protein will be removed from the cereal by successive digestion at a boiling temperature with acid and alkali of a definite strength. The complex body thus obtained is not a definite chemical compound, but may be considered as being composed largely of cellulose.

Use 2 grams of the finely-ground sample and wash on a filter with 5 portions of 10 c.c. each of ether. (The residue from the determination of "ether extract" can be used if desired.)

Transfer the washed material to a 500-c.c. Erlenmeyer flask, add 200 c.c. of boiling 1.25 per cent sulphuric acid, place a funnel in the neck of the flask and boil *gently* for 30 minutes. Filter on a ribbed filter and wash with several portions of boiling water. Transfer the precipitate by means of 200 c.c. of boiling 1.25 per cent sodium hydroxide in a small wash-bottle to the

same 500-c.c. Erlenmeyer flask, and boil again gently for 30 minutes.

Filter on ignited asbestos in a Gooch crucible, wash with boiling water until free from alkali, then with 10 c.c. of alcohol, and finally with 10 c.c. of ether. Dry at the temperature of boiling water to constant weight. Ignite carefully at first, then at a low red heat until the organic matter is destroyed. Calculate the loss on ignition as "crude fibre."

Note. — The filtration will be found to proceed fairly rapidly if the solution is filtered hot and care is taken to keep the residue from the filter as long as possible.

The sulphuric acid and sodium hydroxide should be carefully prepared and the strength determined by titration.

Examination of Malted Cereals. — The relation of the carbohydrates in a malted cereal, which ordinarily consist of maltose, dextrin and starch, may be readily learned by the following simple analytical scheme, due to Sherman.*

Directions. — Mix 5 grams of the ground sample with 125 c.c. of cold water in a 250-c.c. graduated flask and allow it to stand at room temperature for an hour, shaking frequently. Make up to the mark, mix and filter through a dry filter. Determine the reducing sugar in 25 c.c. of the filtrate as described on page 145, and calculate as maltose in the original sample. Measure 50 c.c. of the same filtrate into a 100-c.c. flask, add 5 c.c. of hydrochloric acid (sp. gr. 1.12), and hydrolyze as directed on page 186. Filter and determine the dextrose in the filtrate as on page 145. Subtract the amount due to maltose and calculate the remainder to dextrin by multiplying by 0.9.

Treat another portion of the original sample as described under the determination of starch by acid hydrolysis, page 186, without, however, extracting the soluble carbohydrates. From the dextrose found subtract that given by dextrin and maltose and calculate the remainder to starch.

Notes. — The presence of undissolved material in the flask when diluted to volume renders the result somewhat inaccurate,

* Methods of Organic Analysis, 2d Ed., p. 341.

and the possible presence of other reducing sugars than maltose introduces error, but the results are sufficiently close for comparative tests.

Examination of Fermented Liquors

WINE

General Statements. — The object of a wine analysis is ordinarily to determine whether or not a wine is pure and unadulterated, or whether it has been properly made. Special works furnish sufficient information concerning processes of manufacture, and it is essential to know here only the general composition of the grape-juice or "must" and how, by the natural process of fermentation, this may be altered in the finished product.

The "must" contains *sugars* (mainly dextrose); dextrin; *organic acids and salts*, mainly tartaric and malic acids; salts of inorganic acids, chiefly phosphates, sulphates, and chlorides. Various *extractive matters*, which largely affect the color and flavor of the wine, together with a little *tannin* and albuminous substances, are also present. The wine will contain, then, besides water, the following: *Alcohol*, *glycerine*, frequently some *sugar* that has escaped fermentation, *ethers*, which determine largely the "bouquet" of the wine, and more or less of the acids, salts, coloring and extractive matters of the must, together with varying amounts of carbonic, acetic, and succinic acids.

According to differences in their composition, wines may be divided into various classes, such as "dry" wines, which contain very little sugar, as distinguished from the sweet wines, in which a notable quantity of sugar has escaped fermentation, or to which an addition of sugar has been made subsequent to the main fermentation. Or they may be divided according to the content of alcohol into natural wines and those *fortified* by addition of alcohol, as port, sherry, and madeira.

The composition of the wine may be changed, moreover, by the various methods which are used for its "improvement,"

such as *fortification* already mentioned, *plastering*, *petiotization*, etc. Information regarding these methods will be found in some of the larger works mentioned in the bibliography.

Determinations of value in judging the purity of wine are alcohol, glycerine, extract, ash, total and volatile acids. The actual percentages of these substances are not of so great value as certain relations between them, such as the ratio of ash to extract, extract to alcohol, alcohol to glycerine, alcohol to acids, and volatile to total acids. Examination for preservatives and foreign coloring matters should also be made. It should, perhaps, be stated that the analytical procedure given here is to furnish practice in the examination of a fermented food product, and is by no means as thorough as might be needed to judge the quality or genuineness of a wine.

Specific Gravity. — This is to be taken by means of the pyknometer at 15°.5 C.

Notes. — Where the specific gravity of the sample is known, the various portions taken for analysis can be more conveniently measured than weighed. The results can be calculated to per cent by weight by dividing the results expressed as grams per 100 c.c. by the specific gravity.

Effervescing wines should, before analysis, be vigorously shaken in a large flask to hasten the escape of carbon dioxide. The liquid may then be poured from under the foam into another vessel.

Alcohol. — *Principle.* — The alcohol is obtained freed from everything but water, and its amount determined by ascertaining the specific gravity of the mixture, and taking the per cent from the tables.

Directions. — Measure (or weigh) 100 c.c. of the wine into a 500-c.c. round-bottomed flask. Add 50 c.c. of water and if the wine is very acid a small pinch of precipitated calcium carbonate. With most wines this addition will not be necessary. Distill about 95 c.c. into a 100-c.c. graduated flask. Fill to the mark with distilled water, mix thoroughly, and take the specific gravity of the distillate at 15.5° C. with a pyknometer. The per-

centage of absolute alcohol by volume corresponding to the observed density will be found in Table X, page 217.

To find the alcohol by *weight* in the sample, multiply the per cent of alcohol *by weight* in the distillate as taken from the table, by the weight of the distillate and divide the result by the weight of the sample used.

Notes. — The addition of calcium carbonate is to prevent the distillation of acetic acid. A certain amount of volatile ethers may also pass over into the distillate, but it is so slight that its influence may be neglected.

Normal wines ordinarily contain between 4.5 and 12 per cent of alcohol except in the case of "fortified" wines, where the amount may be even 20 per cent. Fermentation does not yield more than about 14 per cent of alcohol.

Extract. — The method to be employed depends on the proportion of extract. A preliminary calculation should be made by the aid of the formula

$$x = 1 + d - d',$$

where x is the specific gravity of the dealcoholized wine, d the specific gravity of the wine, and d' the specific gravity of the distillate obtained in the determination of alcohol. The value for x is found from Table XI, page 220.

Dry Wines. — (Having an extract content of less than 3 per cent.) Evaporate 50 c.c. on the water-bath to a sirupy consistency in a flat-bottomed platinum dish. Heat the residue in the oven at 100° C. for two hours and a half, cool in a desiccator and weigh.

Sweet Wines. — When the extract content is between 3 and 6 per cent treat 25 c.c. of the sample as described under dry wines. When the amount of extract exceeds 6 per cent it is best to accept the result found from the table and not to determine it gravimetrically.

Notes. — The gravimetric determination will be inaccurate with wines high in extract on account of the serious error caused by drying levulose at high temperatures. The figures in the table are based on determinations made at 75° C. in vacuo.

Wine made from the juice of ripe grapes rarely contains less than 1.5 per cent of extract in the case of white wines and about 2.0 per cent in the case of red wines. The amount of extract decreases of course with age.

Alcohol-extract Ratio. — The municipal laboratory of Paris considers a wine "fortified" if the alcohol exceeds 4.5 times the extract for red wines and 6.5 for white wines. The extract and alcohol should both be expressed in per cent by weight. The amount of added alcohol is calculated by the municipal laboratory by subtracting the "natural" alcohol (extract $\times$ 4.5 or 6.5) from the total alcohol.

Ash. — Ignite the residue from the extract determination as described on page 180.

Note. — The amount of ash in a natural wine averages about 10 per cent of the extract, varying ordinarily between 0.14 per cent and 0.35 per cent.

Glycerine. — The determination of glycerine, and the ratio of glycerine to alcohol is of much value in judging the purity of a wine. The determination of the glycerine, however, is rather difficult and requires some little experience in order to obtain good results. The official method of the Association of Agricultural Chemists will be found in Bur. of Chem., *Bull.* 107, (Rev. Ed.), p. 83. A more accurate modification, however, is that of Ross (*Bull.* 132, p. 85).

Free Acids: Total Acidity Calculated as Tartaric Acid. — Measure 25 c.c. of the wine into a small beaker, heat just below the boiling point to expel carbon dioxide, and titrate with $\frac{N}{10}$ sodium hydroxide and phenolphthalein. In the case of red wines use delicate red litmus paper, taking the end-point when a drop of the liquid placed upon the paper produces a blue spot in the middle of the portion moistened. Calculate the results as tartaric acid. One c.c. $\frac{N}{10}$ sodium hydroxide = 0.0075 gram of tartaric acid.

Volatile Acids Calculated as Acetic Acid. — Measure 50 c.c. of wine into a 300-c.c. flask provided with a cork having two perforations. One is fitted with a tube 6 mm. in diameter and blown out to a bulb 40 mm. in diameter a short distance above the cork; this tube is connected with a condenser. The other perforation carries a tube reaching nearly to the bottom of the flask and drawn out to a small aperture at its lower end; this is connected with a 500-c.c. flask containing water. Heat both flasks to boiling; then lower the flame under that containing the wine, adjusting the flame so that the volume of liquid remains constant, and continue the distillation by means of steam until 200 c.c. have distilled. Titrate the distillate with $\frac{N}{10}$ sodium hydroxide, using phenolphthalein as an indicator. Calculate the results as acetic acid. One c.c. $\frac{N}{10}$ sodium hydroxide = 0.0060 gram of acetic acid.

Hortvet * has described a compact self-contained apparatus for determining the fixed and volatile acids in which the wine is surrounded by boiling water while the steam is being passed through, giving excellent results.

Fixed Acids Calculated as Tartaric Acid. — These may be found by calculating the volatile acids as tartaric and subtracting the result from the total tartaric acid found by direct titration.

Note. — The total acids in a wine vary usually between 0.45 per cent and 1.5 per cent. The acid content is frequently diminished by aging or by the separation of cream of tartar. The volatile acid should, in general, not be over 0.12 to 0.16 per cent, depending upon the age of the wine. A wine properly made should not have the volatile acid, estimated as acetic, exceed one-fourth of the total free acid, calculated as tartaric.

Coloring Matters: Detection of Coal-tar Dyes.† — Fifty c.c. of the sample are diluted to 100 c.c. with water, filtered if neces-

* *J. Ind. Eng. Chem., 1910,* 31.

† Sostegni and Carpentieri: *Ztschr. anal. Chem.,* 35, *1896,* 397.

sary, faintly acidified with hydrochloric or acetic acid, and a piece of white woolen cloth, which has been thoroughly washed with hot water, is immersed in the solution and boiled for five to ten minutes. The cloth is then removed and thoroughly washed with boiling water, and boiled in a dilute solution of ammonia (1 : 50). With some of the dyes the color is stripped from the wool quite readily; with others it is necessary to boil for some time. The wool is removed, the ammoniacal solution made faintly acid with hydrochloric acid, and another piece of white wool is immersed and again boiled. This second dyeing fixes coal-tar dyes on the fibre, but fruit and vegetable colors remain on the first piece of wool.

Notes. — It is absolutely necessary that the second dyeing should be made, as some of the coal-tar dyes will dye a dirty orange in the first acid bath which might be easily passed for vegetable color but on treatment in alkaline bath and second acid bath becomes a bright pink.

Excess of acid should be avoided since some of the colors do not dye readily in strongly acid solution.

Another advantage in the second dyeing is that if a large piece of woolen cloth is used in the first dyeing, and a small piece in the second dyeing, small amounts of coloring matter can be brought out much more decidedly in the second dyeing, where practically all of the vegetable coloring matter has been excluded.

Several colors which are not coal-tar dyes, notably archil and archil derivatives, give reactions by this method and are liable to be confused with coal-tar colors. For hints as to the method for detecting these reference may be made to Bulletin 107, Bureau of Chemistry, page 190.

The further separation and identification of the artificial colors is too difficult a matter to be taken up here. The student is referred for information on this point to the following: Mulliken: The Identification of Commercial Dyestuffs; Loomis: Circular 63, Bureau of Chemistry; Allen: Commercial Organic

Analysis, 4th Ed., Vol. V; Green and others *: The Identification of Dyestuffs on Animal Fibres.

Preservatives. — The preservatives to be sought generally in wines are salicylic and benzoic acids and their salts. Sulphurous acid and sulphites are also used. For methods of detecting other substances less commonly employed, such as abrastol, beta-naphthol, etc., reference may be made to Bulletin 107 of the Bureau of Chemistry. Boric acid is occasionally used, but since a small amount of it is normally present in wines, tests, to be of value, should be quantitative.

Salicylic Acid. — Acidify about 50 c.c. of the wine with 5 c.c. of dilute (1 : 3) sulphuric acid and extract in a separatory funnel with 25 c.c. of ether. Draw off the lower layer, wash the ether twice with water, using 10 c.c. each time and finally evaporate the ether in a porcelain dish at room temperature. To the residue in the dish add 2 to 3 drops of very dilute ferric chloride or better ferric alum solution (App. B). A deep purple or violet color indicates salicylic acid.

Notes. — Not more than 50 c.c. should be used for the test, since a trace of salicylic acid seems normally present in some wines.

The washing with water is to free the ether from traces of sulphuric acid which interferes with the development of the violet color.

Care should be exercised in making the extraction with ether not to shake the separatory funnel too violently, since a troublesome emulsion may result.

Benzoic Acid.† — Acidify about 100 c.c. of wine with sulphuric acid, extract with ether, and evaporate the ethereal solution as in the detection of salicylic acid. Treat the residue with 2 or 3 c.c. of strong sulphuric acid. Heat till white fumes appear; organic matter is charred and benzoic acid is converted into sulpho-benzoic acid. A few crystals of ammonium nitrate are then added. This causes the formation of metadinitrobenzoic

* *J. Soc. Dyers and Colourists, 1905,* 236.

† Mohler: *Bull. Soc. Chim.* [3], **3**, *1890,* 414.

acid. When cool the acid is diluted with water and ammonia added in excess, followed by a drop or two of ammonium sulphide. The nitro-compound becomes converted into ammonium meta-diamidobenzoic acid, which possesses a red color. This reaction takes place immediately, and is seen at the surface of the liquid without stirring.

Sulphurous Acid and Sulphites. — See directions under Beer, page 198.

BEER AND OTHER MALT LIQUORS

Before analysis the sample must be thoroughly shaken in a large flask, in order to remove carbon dioxide.

Specific Gravity. — Taken with a pyknometer at 15.5° C.

Alcohol. — Determined as in the analysis of wine. The addition of calcium carbonate will not be necessary. If the sample foams much this can be prevented by the addition of about half a gram of tannin before distilling.

Extract. — Determine the extract content corresponding to the specific gravity of the dealcoholized beer according to Table XIII. For this purpose employ the formula

$$Sp = g + (1 - g'),$$

in which Sp is the specific gravity of the dealcoholized beer, g the specific gravity of the beer, and g' the specific gravity of the distillate obtained in the determination of alcohol. Instead of using this formula the residue from the distillation of alcohol is sometimes diluted to the original volume, and its specific gravity taken. This is often impracticable owing to the necessity of employing tannic acid to prevent foaming in the distilling flask, and owing to the coagulation of proteids during the distillation.

Note. — The extract of beer cannot be accurately determined by evaporation and drying at the boiling-point of water because of the dehydration of the maltose.

Ash. — Evaporate 25 c.c. to dryness and determine as described on page 180.

Free Acids. — Heat 20 c.c. to incipient boiling to expel carbon dioxide and titrate as in the analysis of wine. Fixed acids, con-

sisting principally of lactic and succinic, are calculated as lactic acid. One c.c. of $\frac{N}{10}$ sodium hydroxide = 0.0090 gram of lactic acid.

Reducing Sugar. — Dilute 25 c.c. of the beer, freed from carbon dioxide, to 100 c.c. Determine the reducing sugar in 25 c.c. of this solution as directed on page 145, enough water being added to make the total volume of the Fehling's solution-sugar mixture 100 c.c. Express the results in terms of maltose, as given in Table XII.

Preservatives. — The preservatives most commonly employed in beer are benzoic and salicylic acids and their sodium salts, sulphites and fluorides.

Benzoic and Salicylic Acids. — Detected as described under Wine.

Sulphites. — Qualitative Test. — Use an apparatus similar to that described for the determination of volatile acids in wine. To 50 c.c. of the sample add about a gram of sodium bicarbonate, 20 c.c. of 20 per cent phosphoric acid, and immediately connect the flask with the condenser. Pass steam through the flask until about 20 c.c. have collected in the distillate. To the distillate add bromine water in slight excess and boil. Expel the excess of bromine and test for sulphuric acid with hydrochloric acid and barium chloride in the usual manner.

Notes. — The method described does not distinguish between free sulphurous acid and that present in the form of sulphites. The former can be distilled without the addition of phosphoric acid.

The presence of sulphites in a sample should not be considered evidence of added preservatives unless an excessive amount is found, since the use of sulphured malt or hops may introduce a small amount. To obtain conclusive data, a quantitative determination of the amount present should be made.

This can be done by a method similar to that used for its detection, distilling in a current of carbon dioxide, absorbing the sulphur dioxide in bromine water and determining the re-

sulting sulphuric acid as barium sulphate. In the case of food products, where sulphides are liable to be present also, the steam should pass through a solution of copper sulphate* before entering the condenser in order to remove any hydrogen sulphide formed by the action of the phosphoric acid. Details of the method will be found in Leach's Food Analysis.

In Bulletin 107 it is recommended to distill into a standard iodine solution and titrate the excess of iodine. This has the disadvantage, however, that other iodine-reducing substances than sulphurous acid may pass into the distillate and give too high results.

Fluorides. — The well-known qualitative test for fluorides by etching a glass plate may be modified by the use of a suitable condenser and made sufficiently delicate to be used here. It is possible also by suitable regulation of the temperature to make the test approximately quantitative.†

Flavoring Extracts

The work on alcoholic liquids can be pleasantly varied by substituting for it, in some cases, the determination of alcohol and other important components of the usual flavoring essences, the most important of which are vanilla and lemon. Several important types of analytical methods, such as the determination of essential oils and quantitative extraction with volatile solvents, are also brought to the attention of the student.

VANILLA

Vanilla extract is a dilute alcoholic tincture of the vanilla bean, the fruit of a climbing plant of the orchid family. The best grades are made by allowing the cut and bruised beans to macerate in the alcohol for several months, the liquid thus obtained being deep brown in color, with a delightful perfume and flavor. Sugar is added to assist in the extraction and to sweeten the product.

* Winton and Bailey: *J. Am. Chem. Soc.*, *1907*, 1499.

† Woodman and Talbot: *J. Am. Chem. Soc.*, *1906*, 1437; *1907*, 1362.

The cost of a quart of the pure extract, according to Winton,* is from about 60 cents to $2.50, depending chiefly upon the grade of beans used.

The composition of five pure vanilla extracts, made from beans of different grades, is given in the following table,† the results being expressed in per cent by weight:

Grade of bean.	Specific gravity.	Vanillin.	Alcohol.	Total residue.	Cane-sugar.
Mexican (whole).....	1.0159	0.125	37.96	22.60	19.90
Mexican (cut).....	1.0146	0.065	39.92	23.10	19.20
South American (whole).	1.1009	0.215	38.58	22.00	19.00
Bourbon (whole).....	1.0166	0.138	38.32	23.13	20.40
Tahiti (whole).....	1.0104	0.108	38.84	21.75	20.00

The adulteration of vanilla extract consists principally in the use of extract of Tonka bean, a cheap substitute somewhat resembling vanilla in its flavor, in the use of artificial preparations of the active principles of vanilla and tonka, vanillin and coumarin, and in the addition of artificial color, usually caramel. A cheap extract may be entirely an artificial mixture, made of artificial vanillin or coumarin, or both, in weak alcohol, colored with caramel. An occasional adulteration is the use of alkali, such as potassium bicarbonate, to hold the resin in solution and permit the use of a more dilute alcohol.

An extract of vanilla of good quality should contain from 25 to 40 per cent of alcohol, from 0.10 to 0.20 per cent of vanillin, and give a good precipitate of vanilla resins. Imitation extracts usually show one or several of the following characteristics: Presence of coumarin; deficiency in resins; abnormally low or high content of vanillin; presence of artificial color; low lead number.

Analytical Methods. — *Alcohol.* — Measure 25 c.c. of the sample, add 100 c.c. of water, and determine the alcohol by

* *Conn. Agr. Exp. Sta. Report, 1901,* 150.
† *Conn. Agr. Exp. Sta. Report, 1901,* 150.

volume, as directed on page 191, omitting the use of calcium carbonate and tannic acid.

Vanillin and Coumarin. — (Modified method of Hess and Prescott).* Weigh 50 grams into a 250-c.c. beaker with marks showing volumes of 80 c.c. and 50 c.c., dilute to 80 c.c., and evaporate to 50 c.c. in a water-bath kept at 70° C. Dilute again to 80 c.c. and evaporate to 50 c.c. Transfer to a 100-c.c. flask, rinsing out the beaker with hot water, add 25 c.c. of lead acetate solution (80 grams of neutral lead acetate made up to a liter), make up to the mark with water, shake and allow it to stand over night. Decant on a small dry filter, pipette off 50 c.c. of the filtrate, and extract it four times in a separatory funnel, using 15 c.c. of ether each time.

Combine the ether extracts in another separatory funnel and wash five times with 2 per cent ammonium hydroxide, using 10 c.c. the first time and 5 c.c. for each subsequent shaking. Set aside the combined ammoniacal solutions for the determination of vanillin.

Transfer the ether solution to a weighed dish and allow the ether to evaporate at room temperature. Dry in a desiccator over sulphuric acid and weigh. If the residue is not white and crystalline stir it for fifteen minutes with 15 c.c. of petroleum ether (boiling point 30° to 40° C.) and decant the clear liquid into a beaker. Repeat the treatment with petroleum ether two or three times. Allow the residue to stand in the air until apparently dry, completing the drying in the desiccator. Weigh, and deduct the weight from the weight of the residue obtained after the ether evaporation, thus obtaining the weight of the *coumarin*. This may be recognized by its characteristic odor, resembling that of "sweet grass," and by Leach's test† as follows: Dissolve the residue in a few drops of hot water, and add one or two drops of $\frac{N}{10}$ iodine in potassium iodide. On stirring with a rod, a brown precipitate will form, which

* *J. Am. Chem. Soc.*, *1905*, 719; *Bur. of Chem.*, *Bull.* 137, 68.

† Leach: "Food Inspection and Analysis," 3d Ed., p. 867.

will gather into dark green flocks. The reaction is especially marked if carried out in a white porcelain crucible or dish.

Slightly acidulate the ammoniacal solution reserved for vanillin with 10 per cent hydrochloric acid. Cool, and shake out in a separatory funnel with four portions of ether, as described for the first ether extraction. Evaporate the ether at room temperature in a weighed dish, dry over sulphuric acid, and weigh the vanillin.

If the residue is white, it may be safely assumed, in the majority of cases, that it is pure vanillin. If dark colored, however, the dry residue should be extracted not less than fifteen times with boiling petroleum ether (boiling point 40° C. or below). Evaporate the solvent, dry and weigh the vanillin. A small amount of the residue, dissolved in two drops of concentrated hydrochloric acid, should give a pink color upon the addition of a crystal of resorcin.

Notes. — The separation of vanillin and coumarin is based on the differences in their chemical constitution. Vanillin is hydroxymethoxybenzoic aldehyde, while coumarin is the anhydride of orthohydroxycinnamic acid. On account of the aldehydic nature of the vanillin, the separation by dilute ammonia is possible, the aldehyde ammonia compound of vanillin being readily soluble in water, while the coumarin remains wholly in the ether.

If a portion of the vanillin, after weighing, be dissolved in two or three drops of ether and allowed to evaporate spontaneously on a microscope slide it shows a characteristic appearance with polarized light. The vanillin crystallizes in slender needles, forming star-shaped clusters. These give a brilliant play of colors with crossed Nicols, even without the selenite plate.

Normal Lead Number. — To a 10-c.c. portion of the filtrate obtained from the lead acetate in the determination of vanillin and coumarin add 25 c.c. of water, sulphuric acid in slight excess, and 100 c.c. of 95 per cent alcohol, let stand over night, filter on a Gooch crucible, wash with alcohol, dry in the oven of the water-bath, ignite for three minutes at low redness, taking care to

avoid the reducing flame, and weigh the lead sulphate. Calculate the normal lead number by the following formula

$$P = \frac{100 \times 0.6831\,(S - W)}{5}$$

in which P = normal lead number, S = grams of lead sulphate corresponding to 2.5 c.c. of the lead acetate solution, as determined from a blank analysis, and W = grams of lead sulphate obtained in 10 c.c. of the filtrate, as just described.

Note. — The normal lead number of genuine vanilla extracts determined by this method ranges from 0.35 to 0.60. Artificial extracts generally are distinctly lower, sometimes as low as 0.03.

More accurate results can be obtained by regulating more closely the time and temperature during the standing of the solution with lead acetate. Winton and Berry* recommend standing 18 hours at 37° to 40° C. They find that, determined in this manner, the minimum normal lead number for vanilla extracts prepared according to the U. S. Pharmacopœia is 0.40.

Resins. — Evaporate 25 or 50 c.c. of the extract to one-third its volume on the water-bath in order to remove the alcohol. Make up to the original volume with hot water. If no alkali has been used in the manufacture of the extract, the resin should appear at this point as a flocculent brown residue. Add acetic acid in slight excess, allow the evaporating-dish to stand in a warm place for a time to separate the resin completely, and filter. Wash the residue on the filter, and save both the filtrate and residue. Test the resin by placing pieces of the filter, with the resin attached, in a few cubic centimeters of dilute caustic potash. The resin is dissolved with a deep red color, and on acidifying is again precipitated. Test the filtrate by adding to it a few drops of basic lead acetate. A bulky precipitate is formed, on account of the organic acid, gums, etc., present.

Confirm the resin test by shaking 5 c.c. portions of the extract in separate test-tubes with 10 c.c. of amyl alcohol and

* *Bur. of Chem., Bull.* 137, 120.

10 c.c. of ether. With pure extracts the upper layers will be colored, varying from light yellow to deep brown; with artificial extracts, free from resin, the amyl alcohol and ether layers will be uncolored.

Note. — While the artificial vanillin, as sold on the market and used in the manufacture of low-grade extracts, is identical with the vanillin of the vanilla bean, it is true that pure extracts owe their value and flavor to other ingredients as well as to the vanillin present. Among these "extractive matters" the resins are important from an analytical standpoint, serving by their presence or absence to determine whether true vanilla is present or the extract entirely artificial. As a quick and ready test, serving to distinguish artificial extracts from genuine preparations of the vanilla bean, the amyl alcohol and ether tests will be found especially useful.

Color: Caramel. — Caramel is the color commonly used in vanilla extracts, although coal-tar dyes have been found. The presence of dyes is sometimes indicated by the color of the amyl alcohol in testing for the resin, they being in many cases soluble in amyl alcohol, but insoluble in ether.

Lead Acetate Test. — The coloring matter present in vanilla extracts is almost completely removed when the dealcoholized extract is treated with a few cubic centimeters of basic lead acetate solution. When caramel is present, the filtrate and precipitate, if any, have the characteristic red-brown color of caramel.

*Marsh Test.** — Evaporate 25 c.c. of the extract until the odor of alcohol is no longer apparent and the liquid is reduced to a thick sirup. Dissolve the residue in water and alcohol, using 26.3 c.c. of 95 per cent alcohol, and making up to volume in a 50-c.c. flask with water. Transfer 25 c.c. of this solution to a separatory funnel; add 25 c.c. of the Marsh reagent and shake, not too vigorously, to avoid emulsification. Allow the layers to separate and repeat the shaking twice more. After the layers have separated clearly, run off the lower layer into a 25-c.c.

* *Bur. of Chem., Bull.* 152, p. 149.

cylinder, and make up to volume with 50 per cent (by volume) alcohol. Filter if necessary and compare in a colorimeter with the remaining 25-c.c. portion (which has not been extracted with the reagent) and express the results as per cent of color insoluble in amyl alcohol.

The Marsh reagent is prepared as follows: Mix 100 c.c. of amyl alcohol, 3 c.c. of sirupy phosphoric acid, and 3 c.c. of water; shake before using. If the reagent becomes colored on standing, the amyl alcohol should be redistilled over 5 per cent phosphoric acid.

Note. — The method is based on the greater solubility in acid amyl alcohol of the natural color of the vanilla bean as compared with caramel. A genuine extract, uncolored with caramel, will not usually show more than 40 per cent of color insoluble in amyl alcohol.

LEMON

Lemon extract is usually made by dissolving oil of lemon, obtained by expression or distillation from the rind of the lemon, in strong alcohol. The product is sometimes colored with the color of lemon peel. The Federal standards * require a content of lemon oil of at least 5 per cent by volume. The expensive ingredient of the extract is the alcohol, since alcohol of at least 80 per cent strength by volume must be used to dissolve 5 per cent of lemon oil; hence in making cheap extracts the manufacturer endeavors to use a dilute alcohol, even under the necessity of omitting a portion or all of the oil of lemon.

The common forms of adulteration of lemon extract are the use of weak alcohol and consequent deficiency of lemon oil, as already noted; the substitution for the lemon oil of small amounts of stronger oils, as oil of citronella, oil of lemon-grass, and the like; the use of *citral*, the odorous principle of lemon oil, used for making the so-called "terpeneless lemon extracts;" and the coloring of the extracts by coal-tar colors or turmeric.

Preliminary Test. — To a little of the extract in a test-tube add seven or eight times its volume of water. A high-grade ex-

* U. S. Dept. Agric., Office of the Secretary, Circ. 19.

tract will show a heavy cloud, due to the precipitation of the lemon oil. If no cloudiness or turbidity appears it may be safely inferred that no oil is present.

Alcohol. — The determination of alcohol is somewhat complicated in this case by the presence of the volatile oil of lemon which must be removed before distilling.

Dilute 20 c.c. of the extract to 100 c.c. with water, and pour the mixture into a dry Erlenmeyer flask containing 5 grams of light magnesium carbonate. Shake thoroughly and filter through a dry filter. Measure 50 c.c. of the clear filtrate, add about 15 c.c. of water, and distill 50 c.c., as directed on page 191. From the specific gravity of the distillate determine the per cent of alcohol by volume, and this, multiplied by 5, will give the percentage in the original extract.

Note. — The magnesia serves to absorb the precipitated oil and prevent it from passing through the filter.

Lemon Oil. — Pipette 20 c.c. of the extract into a Babcock milk bottle; add 1 c.c. dilute hydrochloric acid (1 : 1); then add from 25 to 28 c.c. of water previously warmed to 60° C.; mix and let stand in water at 60° for five minutes; whirl in centrifuge for five minutes; fill with warm water to bring the oil into the graduated neck of the flask; repeat whirling for two minutes; stand the flask in water at 60° C. for a few minutes and read the per cent of oil by volume. If the determination is not made in duplicate the flask should be balanced by another containing an equal weight of water. In case oil of lemon is present in amounts over 2 per cent add to the percentage of oil found 0.4 per cent to correct for the oil retained in solution. If less than 2 per cent and more than 1 per cent is present, add 0.3 per cent for correction.

Color. — Test for coal-tar colors by evaporating a portion of the extract to dryness on the water-bath. Dissolve the residue in water and carry out the double dyeing method, as described on page 194.

It may be advisable not to add any acid to the dye bath, as Naphthol Yellow S, which is commonly used in lemon extracts, dyes wool best from a nearly neutral bath.

To test for turmeric add to a portion of the sample three drops of saturated boric acid solution, *one small drop* of dilute (1 : 10) hydrochloric acid, and a piece of filter-paper so arranged that it is only half immersed in the liquid. Evaporate to dryness on the water-bath. In the presence of turmeric the paper will be colored pink and the test may be confirmed as described on page 154. Excess of hydrochloric acid should be avoided as in testing for boric acid.

To show the presence of natural color derived from lemon peel the following reactions will be found helpful:* Dilute a few cubic centimeters of the extract until the color has nearly disappeared and divide the solution between two test-tubes. To one add a few drops of concentrated hydrochloric acid and to the other a few drops of strong ammonia. In the presence of natural color a distinct yellow color should result in each case.

Citral. — See *Bur. of Chem., Bull.* 137, 70.

* Albrech: *Bur. of Chem., Bull.* 137, 71.

APPENDICES

APPENDIX A

TABLE I

TENSION OF AQUEOUS VAPOR IN MILLIMETERS OF MERCURY FROM 0° TO 30.9° C., REDUCED TO 0° AND SEA-LEVEL

	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
0°	4.57	4.60	4.64	4.67	4.70	4.74	4.77	4.80	4.84	4.87
1	4.91	4.94	4.98	5.02	5.05	5.09	5.12	5.16	5.20	5.23
2	5.27	5.31	5.35	5.39	5.42	5.46	5.50	5.54	5.58	5.62
3	5.66	5.70	5.74	5.78	5.82	5.86	5.90	5.94	5.99	6.03
4	6.07	6.11	6.15	6.20	6.24	6.28	6.33	6.37	6.42	6.46
5	6.51	6.55	6.60	6.64	6.69	6.74	6.78	6.83	6.88	6.92
6	6.97	7.02	7.07	7.12	7.17	7.22	7.26	7.31	7.36	7.42
7	7.47	7.52	7.57	7.62	7.67	7.72	7.78	7.83	7.88	7.94
8	7.99	8.05	8.10	8.15	8.21	8.27	8.32	8.38	8.43	8.49
9	8.55	8.61	8.66	8.72	8.78	8.84	8.90	8.96	9.02	9.08
10	9.14	9.20	9.26	9.32	9.39	9.45	9.51	9.58	9.64	9.70
11	9.77	9.83	9.90	9.96	10.03	10.09	10.16	10.23	10.30	10.36
12	10.43	10.50	10.57	10.64	10.71	10.78	10.85	10.92	10.99	11.06
13	11.14	11.21	11.28	11.36	11.43	11.50	11.58	11.66	11.73	11.81
14	11.88	11.96	12.04	12.12	12.19	12.27	12.35	12.43	12.51	12.59
15	12.67	12.76	12.84	12.92	13.00	13.09	13.17	13.25	13.34	13.42
16	13.51	13.60	13.68	13.77	13.86	13.95	14.04	14.12	14.21	14.30
17	14.40	14.49	14.58	14.67	14.76	14.86	14.95	15.04	15.14	15.23
18	15.33	15.43	15.52	15.62	15.72	15.82	15.92	16.02	16.12	16.22
19	16.32	16.42	16.52	16.63	16.73	16.83	16.94	17.04	17.15	17.26
20	17.36	17.47	17.58	17.69	17.80	17.91	18.02	18.13	18.24	18.35
21	18.47	18.58	18.69	18.81	18.92	19.04	19.16	19.27	19.39	19.51
22	19.63	19.75	19.87	19.99	20.11	20.24	20.36	20.48	20.61	20.73
23	20.86	20.98	21.11	21.24	21.37	21.50	21.63	21.76	21.89	22.02
24	22.15	22.29	22.42	22.55	22.69	22.83	22.96	23.10	23.24	23.38
25	23.52	23.66	23.80	23.94	24.08	24.23	24.37	24.52	24.66	24.81
26	24.96	25.10	25.25	25.40	25.55	25.70	25.86	26.01	26.16	26.32
27	26.47	26.63	26.78	26.94	27.10	27.26	27.42	27.58	27.74	27.90
28	28.07	28.23	28.39	28.56	28.73	28.89	29.06	29.23	29.40	29.57
29	29.74	29.92	30.09	30.26	30.44	30.62	30.79	30.97	31.15	31.33
30	31.51	31.69	31.87	32.06	32.24	32.43	32.61	32.80	32.99	33.18

TABLE II

WEIGHT IN MILLIGRAMS OF A CUBIC CENTIMETER OF CARBON DIOXIDE FROM 746 TO 778 MILLIMETERS PRESSURE AND FROM 10° TO 25° C. CORRECTED FOR THE TENSION OF AQUEOUS VAPOR

(Extended from the Tables of Dietrich*)

	Millimeters.																	
	746	748	750	752	754	756	758	760	762	764	766	768	770	772	774	776	778	
10°	1.83936	1.84435	1.84934	1.85433	1.85933	1.86432	1.86931	1.87430	1.87930	1.88429	1.88928	1.89427	1.89926	1.90425	1.90924	1.91423	1.91922	10°
11	1.83134	1.83631	1.84129	1.84626	1.85123	1.85621	1.86118	1.86616	1.87113	1.87610	1.88108	1.88605	1.89103	1.89601	1.90098	1.90596	1.91094	11
12	1.82324	1.82820	1.83315	1.83811	1.84307	1.84802	1.85298	1.85793	1.86289	1.86785	1.87280	1.87776	1.88271	1.88767	1.89263	1.89758	1.90253	12
13	1.81521	1.82015	1.82510	1.83004	1.83499	1.83993	1.84488	1.84982	1.85477	1.85971	1.86466	1.86960	1.87455	1.87950	1.88444	1.88939	1.89343	13
14	1.80714	1.81208	1.81701	1.82194	1.82687	1.83181	1.83674	1.84167	1.84661	1.85154	1.85647	1.86141	1.86634	1.87127	1.87620	1.88113	1.88607	14
15	1.79880	1.80371	1.80861	1.81352	1.81843	1.82333	1.82824	1.83314	1.83805	1.84296	1.84786	1.85277	1.85767	1.86257	1.86748	1.87239	1.87729	15
16	1.79056	1.79545	1.80034	1.80523	1.81012	1.81501	1.81990	1.82479	1.82968	1.83457	1.83946	1.84435	1.84924	1.85413	1.85902	1.86391	1.86880	16
17	1.78216	1.78703	1.79189	1.79676	1.80163	1.80650	1.81136	1.81623	1.82110	1.82596	1.83083	1.83570	1.84056	1.84543	1.85030	1.85516	1.86003	17
18	1.77381	1.77867	1.78353	1.78838	1.79324	1.79809	1.80295	1.80781	1.81266	1.81752	1.82337	1.82723	1.83209	1.83694	1.84180	1.84666	1.85151	18
19	1.76530	1.77014	1.77498	1.77982	1.78466	1.78950	1.79434	1.79917	1.80401	1.80885	1.81369	1.81853	1.82337	1.82821	1.83305	1.83789	1.84273	19
20	1.75681	1.76113	1.76645	1.77127	1.77610	1.78092	1.78574	1.79056	1.79538	1.80021	1.80503	1.80985	1.81467	1.81949	1.82432	1.82914	1.83396	20
21	1.74818	1.75299	1.75780	1.76260	1.76741	1.77221	1.77702	1.78183	1.78663	1.79144	1.79624	1.80105	1.80586	1.81066	1.81547	1.82028	1.82508	21
22	1.73949	1.74428	1.74907	1.75386	1.75865	1.76344	1.76823	1.77302	1.77781	1.78260	1.78739	1.79218	1.79697	1.80176	1.80655	1.81134	1.81613	22
23	1.73068	1.73546	1.74023	1.74501	1.74978	1.75455	1.75933	1.76410	1.76888	1.77365	1.77842	1.78320	1.78797	1.79274	1.79752	1.80229	1.80706	23
24	1.72178	1.72654	1.73129	1.73605	1.74081	1.74556	1.75032	1.75508	1.75984	1.76459	1.76935	1.77411	1.77886	1.78362	1.78838	1.79314	1.79789	24
25	1.71277	1.71751	1.72225	1.72699	1.73173	1.73648	1.74122	1.74596	1.75070	1.75544	1.76018	1.76492	1.76967	1.77441	1.77915	1.78389	1.78863	25

* Ztschr. anal. Chem., 4 (1865), 141

TABLE III

RELATIVE HUMIDITY

Air temp.	Depression of wet-bulb thermometer.																				
	0.5	1.0	2.0	3.0	4.0	5.0	6.0	7.0	8.0	9.0	10.0	11.0	12.0	13.0	14.0	15.0	16.0	17.0	18.0	19.0	20.0	21.0
20	92	85	70	55	40	26	12	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....
25	94	87	74	62	49	37	25	13	1	....	....	....	....	....	....	....	....	....	....	....	....	....
30	94	89	78	67	56	46	36	26	16	6	....	....	....	....	....	....	....	....	....	....	....	....
35	95	91	81	72	63	54	45	36	27	19	10	2	....	....	....	....	....	....	....	....	....	....
40	96	92	83	75	68	60	52	45	37	29	22	15	7	0	....	....	....	....	....	....	....	....
45	96	93	86	78	71	64	57	51	44	38	31	25	18	12	6	....	....	....	....	....	....	....
50	96	93	87	80	74	67	61	55	49	43	38	32	27	21	16	10	5	0	....	....	....	....
55	97	94	88	82	76	70	65	59	54	49	43	38	33	28	23	19	14	9	5	0	....	....
60	97	94	89	83	78	73	68	63	58	53	48	43	39	34	30	26	21	17	13	9	5	1
65	97	95	90	85	80	75	70	66	61	56	52	48	44	39	35	31	27	24	20	16	12	9
70	98	95	90	86	81	77	72	68	64	59	55	51	48	44	40	36	33	29	25	22	19	15
75	98	96	91	86	82	78	74	70	66	62	58	54	51	47	44	40	37	34	30	27	24	21
80	98	96	91	87	83	79	75	72	68	64	61	57	54	50	47	44	41	38	35	32	29	26
85	....	96	92	88	84	70	76	73	69	66	62	59	56	52	49	46	43	41	38	35	32	30
90	....	96	92	89	85	81	78	74	71	68	65	61	58	55	52	49	47	44	41	39	36	34
95	....	96	93	89	85	82	79	75	72	69	66	63	60	57	54	51	49	46	43	41	38	36
100	....	96	93	89	86	83	80	77	73	70	68	65	62	59	56	54	51	49	46	44	41	39

Reduced from Weather Bureau Bulletin No. 235.

TABLE IV

TABLE OF AVERAGE COMPOSITION OF WATERS. BASED ON MASSACHUSETTS DATA

(Parts per million)

Class.	Turbidity and sediment.	Odor.	Color*	Alb. ammonia.	Free ammonia.	Nitrites.	Nitrates.	Chlorine.	Hardness.	Total solids.
Meteoric water (rain and snow)	Slight to considerable.	None to tarry.	0.00	0.010 to 0.150	0.100 to 0.400	0.000 to 0.003	0.000 to 0.050	0.3 to 3.0	0.0	2.0 to 10.0
Water of brooks and ponds	Slight to considerable.	Vegetable to grassy.	0.10 to 2.00	0.100 to 0.400	0.000 to 0.020	0.000 to 0.002	0.000 to 0.100	0.0 to 6.0	0.0 to 20.0	20.0 to 30.0
Unpolluted springs or ground-waters	None.	None.	0.00	0.000 to 0.010	0.000	0.000	0.100 to 0.400	1.0 to 6.0	3.0 to 25.0	20.0 to 40.0
Sewage	Decided milky to thick dirty.	Offensive.	0.3 to 0.7	1.50 to 4.50	10.000 to 20.000	0.000 to 0.500	0.000 to 0.100	30.0 to 60.0	30.0 to 50.0	250.0 to 450.0
Sand-filtered effluent	None.	None.	0.00	0.020 to 0.100	0.000 to 0.050	0.000 to 0.002	5.000 to 15.000	30.0 to 60.0	35.0 to 55.0	150.0 to 250.0
Polluted rivers	Distinct to decided.	Musty to offensive.	0.4	0.286 to 1.200	0.210 to 0.500	0.010 to 0.060	0.300 to 0.550	9.7 to 18.0	40.0 to 50.0	77.0 to 90.0
SPECIAL EXAMPLES										
Polluted wells, No. 1	None.	None.	0.00	0.034	0.182	0.004	10.000	34.4	94.0	279.0
Polluted wells, No. 2	None.	Dist. mouldy.	0.00	0.080	5.760	0.020	7.000	28.1	36.0	166.0
Polluted wells, No. 3	Milky.	Dec. musty and disagreeable.	0.10	0.220	16.800	0.001	0.250	14.4	25.0	120.0
Purified ground-waters, No. 1	None.	None.	0.00	0.062	0.000	0.000	16.665	29.0	91.0	224.0
Purified ground-waters, No. 2	None.	None.	0.00	0.004	0.000	0.000	5.000	20.0	130.0	250.0

* Figures for color in all tables are given in the Nessler standard.

TABLE V

NORMAL WATERS FROM VARIOUS PARTS OF NORTH AMERICA

(Parts per million)

No.		Color.	Total residue.	Ammonia.		Nitrogen as		Chlorine.	Hardness.	Ox. cons.	Iron.
				Albuminoid.	Free.	Nitrites.	Nitrates.				
1	Brook, Nova Scotia	2.20	58.0	0.262	0.012	0.000	0.020	....	6.0	....	....
2	Spring, off coast of Maine	0.0	111.0	0.018	0.000	0.000	0.380	16.7	50.0	0.234	....
3	Spring, Mooselauke, N. H.	0.0	25.5	0.034	0.000	0.000	0.010	0.5	13.0	....	....
4	Small stream, Vermont	0.05	188.5	0.040	0.016	0.000	0.030	0.8	191.0	0.780	....
5	Cistern, Massachusetts	0.05	30.5	0.136	0.032	0.000	0.030	2.5	17.0	.....	....
6	Driven well, coast of Massachusetts	1.2	110.0	0.120	0.380	0.001	0.000	24.0	50.0	1.026	1.63
7	Driven well, coast of Massachusetts	0.5	54.0	0.028	0.002	0.000	0.000	4.7	28.0	0.390	....
8	Spring, Central Massachusetts	0.0	54.0	0.004	0.000	0.000	0.020	1.8	22.0	0.000	....
9	Small stream, New York	0.42	31.5	0.070	0.000	0.000	0.070	0.8	6.0	....	....
10	Lake, Adirondack Mts.	0.12	24.0	0.064	0.000	0.000	0.000	0.4	15.0	....	....
11	Reservoir, Newark, N. J.	0.18	47.0	0.116	0.002	0.001	0.200	2.0	24.0	....	....
12	Driven well, South Carolina	0.0	55.0	0.046	0.010	0.000	0.600	2.2	17.0	....	....
13	Spring, Georgia	0.15	123.0	0.052	0.014	0.001	0.100	1.8	112.0	....	....
14	Mississippi River, in Missouri	0.25	filtered 24.8	0.546	0.008	0.010	0.580	7.3	113.0	....	....
15	Deep well near Lake Superior	0.0	186.0	0.006	0.200	0.000	0.030	54.0	80.0	....	....
16	Missouri River, in Montana	0.08	261.0	0.020	0.008	0.000	0.130	13.5	154.0	....	....
17	River Sac, Missouri	0.05	210.0	0.070	0.032	0.007	0.500	2.0	156.0	....	....
18	Assiniboine, Winnipeg	0.08	586.0	0.206	0.000	0.000	0.000	24.5	406.0	....	....
19	Deep well, Lake Winnipeg	0.0	1070.0	0.034	0.000	0.001	1.600	234.2	550.0	....	....
20	Deep well, Texas	0.0	566.0	0.020	0.194	0.000	0.000	101.0	22.0	....	....
21	Cistern, Jamaica	0.33	.....	0.040	0.012	0.000	0.100	1.4	5.4	....	....

TABLE VI

SEWAGE AND POLLUTED WATERS FROM VARIOUS PARTS OF NORTH AMERICA

(Parts per million)

No.		Color.	Total residue.	Ammonia.		Nitrogen as		Chlorine.	Hardness.	Ox. cons.
				Albuminoid.	Free.	Nitrites.	Nitrates.			
1	Illinois and Michigan Canal, Lockport, Ill..... (Streams Examination, Chicago, 1903.)	....	630.00	3.600	16.000	0.000	0.100	93.9	....	....
2	Desplaines River, South Joliet..... (Streams Examination, Chicago, 1903.)	0.15	282.0	0.960	3.000	0.072	0.050	30.5	....	9.9
3	Illinois River at Morris..... (Chem. Survey of Waters of Illinois, 1897–1902.)	....	679.02	2.400	9.200	0.060	0.350	92.0	....	29.5
4	Illinois River at Beardstown..... (Streams Examination, Chicago, 1903.)	0.23	392.0	0.408	2.800	0.046	2.000	28.0	....	6.8
5	Mississippi River, New Orleans..... (Report on Water and Sewerage, New Orleans, 1903.)	....	580.0	0.290	0.013	0.023	0.560	7.5	....	8.8
6	Ohio River..... (Investigations into the Purification of the Ohio River for City of Cincinnati, 1899.)	0.08	1057.0	0.746	0.018	0.004	1.020	6.0	....	14.3
7	Inlet to Easton's Pond..... (Board of Health, Newport, R. I.)	1.000	121.0	0.790	0.520	0.066	0.500	12.2	25.0	10.8
8	Port Townsend, Glendale Dairy..... (Washington Geol. Survey, Vol. I, Part V.)	Clear	239.0	0.187	0.200	0.000	6.940	46.7	146.4	....
9	Parral. From Carafe on Table..... (Potable Waters of Mexico. Trans. Amer. Inst. Mining Engineers, Nov., 1901.)	0.00	566.0	0.080	0.000	0.040	15.000	89.7	224.0	....
10	Well, McConway-Torley Mfg Co., Pittsburgh.. (Filtration Commission, Pittsburgh, 1899.)	1.50	320.0	0.124	0.018	0.000	1.500	30.1	160.0	....
11	Well, Sligo Mills, Pittsburgh..... (Filtration Commission, Pittsburgh, 1899.)	....	306.0	0.110	0.230	0.040	0.000	18.4	144.0	....

TABLE VI — *Continued*

SEWAGE AND POLLUTED WATERS

(Parts per million)

No.		Color.	Total residue.	Ammonia.		Nitrogen as		Chlorine.	Hardness.	Ox. cons.
				Albuminoid.	Free.	Nitrites.	Nitrates.			
1	Sewage, manufacturing city	....	663.0	7.60	55.00	0.000	0.000	93.5	....	71.00
2	Sewage, small town	....	480.0	4.60	16.40	0.160	1.220	55.8	....	56.00
3	Sewage, dilute	....	290.0	2.960	6.400	0.640	0.020	37.2	....	27.80
4	Brook, Massachusetts	....	.....	0.154	0.034	0.030	2.750	15.6	50.0	....
5	River, Massachusetts	....	73.9	0.214	0.873	0.002	.....	5.2	....	....
6	Brook, below filter-beds	0.25	221.7	0.262	3.547	0.209	3.127	46.7	53.0	4.30
7	Well, coast Massachusetts, May	0.0	.....	0.044	0.056	0.001	1.750	44.0	32.0	....
8	Well, coast Massachusetts, July	0.0	.....	0.040	0.394	0.150	10.00	54.0	35.0	....
9	Well, Newport, R. I.	0.0	150.0	0.004	0.004	0.000	3.900	26.0	57.0	0.000
10	Well, Essex, Mass.	0.02	583.0	0.100	0.032	0.005	3.500	110.0	194.4	....
11	Well, Framingham, Mass.	0.0	691.0	0.080	0.930	0.010	50.00	72.2	172.2	....
12	Well, Roxbury, Mass.	0.16	972.0	0.104	0.068	0.027	46.00	108.6	225.2	4.130

TABLE VII

SULPHATES IN WATER

(Reduced from table in article by H. F. Muer, J. Ind. Eng. Chem., 1911, Vol. 3, p. 553)

Depth, cm.	SO_3, pts. per million.	Depth, cm.	SO_3, pts. per million.	Depth, cm.	SO_3, pts. per million.	Depth, cm.	SO_3, pts. per million.
1.5	313.0	6.7	72.0	11.9	47.3	17.1	37.3
1.6	280.0	6.8	71.3	12.0	47.0	17.2	37.3
1.7	250.0	6.9	70.5	12.1	46.8	17.3	37.0
1.8	238.0	7.0	69.8	12.2	46.5	17.4	36.8
1.9	225.0	7.1	69.0	12.3	46.3	17.5	36.8
2.0	213.0	7.2	68.3	12.4	46.0	17.6	36.5
2.1	200.0	7.3	67.5	12.5	45.8	17.7	36.3
2.2	190.0	7.4	66.8	12.6	45.5	17.8	36.0
2.3	183.0	7.5	66.0	12.7	45.3	17.9	36.0
2.4	175.0	7.6	65.3	12.8	45.0	18.0	35.8
2.5	168.0	7.7	64.8	12.9	44.8	18.1	35.8
2.6	163.0	7.8	64.0	13.0	44.5	18.2	35.5
2.7	158.0	7.9	63.5	13.1	44.3	18.3	35.3
2.8	153.0	8.0	62.8	13.2	44.0	18.4	35.3
2.9	148.0	8.1	62.3	13.3	43.8	18.5	35.0
3.0	143.0	8.2	61.8	13.4	43.5	18.6	35.0
3.1	138.0	8.3	61.0	13.5	43.3	18.7	34.8
3.2	135.0	8.4	60.5	13.6	43.3	18.8	34.5
3.3	130.0	8.5	60.0	13.7	43.0	18.9	34.5
3.4	128.0	8.6	59.5	13.8	42.8	19.0	34.3
3.5	125.0	8.7	59.0	13.9	42.5	19.1	34.3
3.6	122.5	8.8	58.5	14.0	42.5	19.2	34.0
3.7	120.0	8.9	58.0	14.1	42.3	19.3	33.8
3.8	117.5	9.0	57.5	14.2	42.0	19.4	33.8
3.9	115.0	9.1	57.0	14.3	41.8	19.5	33.5
4.0	112.5	9.2	56.5	14.4	41.5	19.6	33.5
4.1	110.0	9.3	56.3	14.5	41.5	19.7	33.3
4.2	107.5	9.4	55.8	14.6	41.3	19.8	33.0
4.3	105.0	9.5	55.3	14.7	41.0	19.9	33.0
4.4	102.5	9.6	54.8	14.8	40.8	20.0	32.8
4.5	100.0	9.7	54.5	14.9	40.5	20.1	32.5
4.6	98.3	9.8	54.0	15.0	40.5	20.2	32.5
4.7	96.5	9.9	53.8	15.1	40.3	20.3	32.3
4.8	94.8	10.0	53.3	15.2	40.0	20.4	32.0
4.9	93.0	10.1	52.8	15.3	40.0	20.5	32.0
5.0	91.5	10.2	52.5	15.4	39.8	20.6	31.8
5.1	90.0	10.3	52.3	15.5	39.8	20.7	31.5
5.2	88.5	10.4	51.8	15.6	39.5	20.8	31.5
5.3	87.3	10.5	51.5	15.7	39.3	20.9	31.3
5.4	85.8	10.6	51.0	15.8	39.3	21.0	31.3
5.5	84.5	10.7	50.8	15.9	39.0	21.1	31.0
5.6	83.3	10.8	50.5	16.0	39.0	21.2	30.8
5.7	82.0	10.9	50.3	16.1	38.8	21.3	30.8
5.8	81.0	11.0	50.0	16.2	38.5	21.4	30.5
5.9	80.0	11.1	49.5	16.3	38.5	21.5	30.3
6.0	78.8	11.2	49.3	16.4	38.3	21.6	30.3
6.1	77.8	11.3	48.8	16.5	38.3	21.7	30.0
6.2	76.8	11.4	48.5	16.6	38.0	21.8	30.0
6.3	75.8	11.5	48.3	16.7	38.0	21.9	29.8
6.4	74.8	11.6	48.0	15.8	37.8	22.0	29.5
6.5	73.8	11.7	47.8	16.9	37.5	....	....
6.6	73.0	11.8	47.5	17.0	37.5	....	....

TABLE VIII

TABLE OF HARDNESS, SHOWING THE PARTS OF CALCIUM CARBONATE ($CaCO_3$) IN 1,000,000 FOR EACH TENTH OF A CUBIC CENTIMETER OF SOAP SOLUTION USED

	0.0 cu. cm.	0.1 cu. cm.	0.2 cu. cm.	0.3 cu. cm.	0.4 cu. cm.	0.5 cu. cm.	0.6 cu. cm.	0.7 cu. cm.	0.8 cu. cm.	0.9 cu. cm.
0.0	....	....	....	....	....	....	....	0.0	1.6	3.2
1.0	4.8	6.3	7.9	9.5	11.1	12.7	14.3	15.6	16.9	18.2
2.0	19.5	20.8	22.1	23.4	24.7	26.0	27.3	28.6	29.9	31.2
3.0	32.5	33.8	35.1	36.4	37.7	39.0	40.3	41.6	42.9	44.3
4.0	45.7	47.1	48.6	50.0	51.4	52.9	54.3	55.7	57.1	58.6
5.0	60.0	61.4	62.9	64.3	65.7	67.1	68.6	70.0	71.4	72.9
6.0	74.3	75.7	77.1	78.6	80.0	81.4	82.9	84.3	85.7	87.1
7.0	88.6	90.0	91.4	92.9	94.3	95.7	97.1	98.6	100.0	101.5
8.0	103.0	104.5	106.0	107.5	109.0	110.5	112.0	113.5	115.0	116.5
9.0	118.0	119.5	121.1	122.6	124.1	125.6	127.1	128.6	130.1	131.6
10.0	133.1	134.6	136.1	137.6	139.1	140.6	142.1	143.7	145.2	146.8
11.0	148.4	150.0	151.6	153.2	154.8	156.3	157.9	159.5	161.1	162.7
12.0	164.3	165.9	167.5	169.0	170.6	172.2	173.8	175.4	177.0	178.6
13.0	180.2	181.7	183.3	184.9	186.5	188.1	189.7	191.3	192.9	194.4
14.0	196.0	197.6	199.2	200.8	202.4	204.0	205.6	207.1	208.7	210.3
15.0	211.9	213.5	215.1	216.8	218.5	220.2	221.8	223.5	225.2	226.9

TABLE IX

FOR CORRECTING THE SPECIFIC GRAVITY OF MILK ACCORDING TO TEMPERATURE ADAPTED FROM THE TABLE OF VIETH

(Temperature in Degrees Centigrade)

Specific gravity.	10°	11°	12°	13°	14°	15°	16°	17°	18°	19°	20°
1.025	24.1	24.3	24.5	24.6	24.7	24.9	25.1	25.3	25.4	25.6	25.9
26	25.1	25.2	25.4	25.5	25.7	25.9	26.1	26.3	26.5	26.7	27.0
27	26.1	26.2	26.4	26.5	26.7	26.9	27.1	27.4	27.5	27.7	28.0
28	27.0	27.2	27.4	27.5	27.7	27.9	28.1	28.4	28.5	28.7	29.0
29	28.0	28.2	28.4	28.5	28.7	28.9	29.1	29.4	29.5	29.8	30.1
30	29.0	29.1	29.3	29.5	29.7	29.9	30.1	30.4	30.5	30.8	31.1
31	29.9	30.1	30.3	30.4	30.6	30.9	31.2	31.4	31.5	31.8	32.2
32	30.9	31.1	31.3	31.4	31.6	31.9	32.2	32.4	32.6	32.9	33.2
33	31.8	32.0	32.3	32.4	32.6	32.9	33.2	33.4	33.6	33.9	34.2
34	32.7	33.0	33.2	33.4	33.6	33.9	34.2	34.4	34.6	34.9	35.2
35	33.6	33.9	34.1	34.4	34.6	34.9	35.2	35.4	35.6	35.9	36.2

Directions. — Find the observed gravity in the left-hand column. Then, in the same line, and under the observed temperature, will be found the corrected reading.

TABLE X

PERCENTAGE OF ALCOHOL FROM THE SPECIFIC GRAVITY AT 15°.5 C. (HEHNER)

Sp. gr. 15°.5 C.	Per cent alcohol by weight.	Per cent alcohol by volume.	Sp. gr. 15°.5 C.	Per cent alcohol by weight.	Per cent alcohol by volume.	Sp. gr. 15°.5 C.	Per cent alcohol by weight.	Per cent alcohol by volume.
1.0000	0.00	0.00						
0.9999	0.05	0.07	0.9959	2.33	2.93	0.9919	4.69	5.86
8	0.11	0.13	8	2.39	3.00	8	4.75	5.94
7	0.16	0.20	7	2.44	3.07	7	4.81	6.02
6	0.21	0.26	6	2.50	3.14	6	4.87	6.10
5	0.26	0.33	5	2.56	3.21	5	4.94	6.17
4	0.32	0.40	4	2.61	3.28	4	5.00	6.24
3	0.37	0.46	3	2.67	3.35	3	5.06	6.32
2	0.42	0.53	2	2.72	3.42	2	5.12	6.40
1	0.47	0.60	1	2.78	3.49	1	5.19	6.48
0	0.53	0.66	0	2.83	3.55	0	5.25	6.55
0.9989	0.58	0.73	0.9949	2.89	3.62	0.9909	5.31	6.63
8	0.63	0.79	8	2.94	3.69	8	5.37	6.71
7	0.68	0.86	7	3.00	3.76	7	5.44	6.78
6	0.74	0.93	6	3.06	3.83	6	5.50	6.86
5	0.79	0.99	5	3.12	3.90	5	5.56	6.94
4	0.84	1.06	4	3.18	3.98	4	5.62	7.01
3	0.89	1.13	3	3.24	4.05	3	5.69	7.09
2	0.95	1.19	2	3.29	4.12	2	5.75	7.17
1	1.00	1.26	1	3.35	4.20	1	5.81	7.25
0	1.06	1.34	0	3.41	4.27	0	5.87	7.32
0.9979	1.12	1.42	0.9939	3.47	4.34	0.9899	5.94	7.40
8	1.19	1.49	8	3.53	4.42	8	6.00	7.48
7	1.25	1.57	7	3.59	4.49	7	6.07	7.57
6	1.31	1.65	6	3.65	4.56	6	6.14	7.66
5	1.37	1.73	5	3.71	4.63	5	6.21	7.74
4	1.44	1.81	4	3.76	4.71	4	6.28	7.83
3	1.50	1.88	3	3.82	4.78	3	6.36	7.92
2	1.56	1.96	2	3.88	4.85	2	6.43	8.01
1	1.62	2.04	1	3.94	4.93	1	6.50	8.10
0	1.69	2.12	0	4.00	5.00	0	6.57	8.18
0.9969	1.75	2.20	0.9929	4.06	5.08	0.9889	6.64	8.27
8	1.81	2.27	8	4.12	5.16	8	6.71	8.36
7	1.87	2.35	7	4.19	5.24	7	6.78	8.45
6	1.94	2.43	6	4.25	5.32	6	6.86	8.54
5	2.00	2.51	5	4.31	5.39	5	6.93	8.63
4	2.06	2.58	4	4.37	5.47	4	7.00	8.72
3	2.11	2.62	3	4.44	5.55	3	7.07	8.80
2	2.17	2.72	2	4.50	5.63	2	7.13	8.88
1	2.22	2.79	1	4.56	5.71	1	7.20	8.96
0	2.28	2.86	0	4.62	5.78	0	7.27	9.04

TABLE X. — (*Continued*)

PERCENTAGE OF ALCOHOL

Sp. gr. 15°.5 C.	Per cent alcohol by weight.	Per cent alcohol by volume.	Sp. gr. 15°.5 C.	Per cent alcohol by weight.	Per cent alcohol by volume.	Sp. gr. 15°.5 C.	Per cent alcohol by weight.	Per cent alcohol by volume.
0.9879	**7.33**	**9.13**	5	10.46	12.96	2	13.77	16.98
8	7.40	9.21	4	10.54	13.05	1	13.85	17.08
7	7.47	9.29	3	10.62	13.15	0	13.92	17.17
6	7.53	9.37	2	10.69	13.24			
5	7.60	9.45	1	10.77	13.34	**0.9789**	**14.00**	**17.26**
4	7.67	9.54	0	10.85	13.43	8	14.09	17.37
3	7.73	9.62				7	14.18	17.48
2	7.80	9.70	**0.9829**	**10.92**	**13.52**	6	14.27	17.59
1	7.87	9.78	8	11.00	13.62	5	14.36	17.70
0	7.93	9.86	7	11.08	13.72	4	14.45	17.81
			6	11.15	13.81	3	14.55	17.92
0.9869	**8.00**	**9.95**	5	11.23	13.90	2	14.64	18.03
8	8.07	10.03	4	11.31	13.99	1	14.73	18.14
7	8.14	10.12	3	11.38	14.09	0	14.82	18.25
6	8.21	10.21	2	11.46	14.18			
5	8.29	10.30	1	11.54	14.27	**0.9779**	**14.90**	**18.36**
4	8.36	10.38	0	11.62	14.37	8	15.00	18.48
3	8.43	10.47				7	15.08	18.58
2	8.50	10.56	**0.9819**	**11.69**	**14.46**	6	15.17	18.68
1	8.57	10.65	8	11.77	14.56	5	15.25	18.78
0	8.64	10.73	7	11.85	14.65	4	15.33	18.88
			6	11.92	14.74	3	15.42	18.98
0.9859	**8.71**	**10.82**	5	12.00	14.84	2	15.50	19.08
8	8.79	10.91	4	12.08	14.93	1	15.58	19.18
7	8.86	11.00	3	12.15	15.02	0	15.67	19.28
6	8.93	11.08	2	12.23	15.12			
5	9.00	11.17	1	12.31	15.21	**0.9769**	**15.75**	**19.39**
4	9.07	11.26	0	12.38	15.30	8	15.83	19.49
3	9.14	11.35				7	15.92	19.59
2	9.21	11.44	**0.9809**	**12.46**	**15.40**	6	16.00	19.68
1	9.29	11.52	8	12.54	15.49	5	16.08	19.78
0	9.36	11.61	7	12.62	15.58	4	16.15	19.87
			6	12.69	15.68	3	16.23	19.96
0.9849	**9.43**	**11.70**	5	12.77	15.77	2	16.31	20.06
8	9.50	11.79	4	12.85	15.86	1	16.38	20.15
7	9.57	11.87	3	12.92	15.96	0	16.46	20.24
6	9.64	11.96	2	13.00	16.05			
5	9.71	12.05	1	13.08	16.15	**0.9759**	**16.54**	**20.33**
4	9.79	12.13	0	13.15	16.24	8	16.62	20.43
3	9.86	12.22				7	16.69	20.52
2	9.93	12.31	**0.9799**	**13.23**	**16.33**	6	16.77	20.61
1	10.00	12.40	8	13.31	16.43	5	16.85	20.71
0	10.08	12.49	7	13.38	16.52	4	16.92	20.80
			6	13.46	16.61	3	17.00	20.89
0.9839	**10.15**	**12.58**	5	13.54	16.70	2	17.08	20.99
8	10.23	12.68	4	13.62	16.80	1	17.17	21.09
7	10.31	12.77	3	13.69	16.89	0	17.25	21.19
6	10.38	12.87						

TABLE X. — (*Continued*)

PERCENTAGE OF ALCOHOL

Sp. gr. 15°.5 C.	Per cent alcohol by weight.	Per cent alcohol by volume.	Sp. gr. 15°.5 C.	Per cent alcohol by weight.	Per cent alcohol by volume.	Sp. gr. 15°.5C.	Per cent alcohol by weight.	Per cent alcohol by volume.
0.9749	**17.33**	**21.29**	6	20.00	24.48	3	22.62	27.59
8	17.42	20.39	5	20.08	24.58	2	22.69	27.68
7	17.50	21.49	4	20.17	24.68	1	22.77	27.77
6	17.58	21.59	3	20.25	24.78	0	22.85	27.86
5	17.67	21.69	2	20.33	24.88			
4	17.75	21.79	1	20.42	24.98	**0.9679**	**22.92**	**27.95**
3	17.83	21.89	0	20.50	25.07	8	23.00	28.04
2	17.92	21.99				7	23.08	28.13
1	18.00	22.09	**0.9709**	**20.58**	**25.17**	6	23.15	28.22
0	18.08	22.18	8	20.67	25.27	5	23.23	28.31
			7	20.75	25.37	4	23.31	28.41
0.9739	**18.15**	**22.27**	6	20.83	25.47	3	23.38	28.50
8	18.23	22.36	5	20.92	25.57	2	23.46	28.59
7	18.31	22.46	4	21.00	25.67	1	23.54	28.68
6	18.38	22.55	3	21.08	25.76	0	23.62	28.77
5	18.46	22.64	2	21.15	25.86			
4	18.54	22.73	1	21.23	25.95	**0.9669**	**23.69**	**28.86**
3	18.62	22.82	0	21.31	26.04	8	23.77	28.95
2	18.69	22.92				7	23.85	29.04
1	18.77	23.01	**0.9699**	**21.38**	**26.13**	6	23.92	29.13
0	18.85	23.10	8	21.46	26.22	5	24.00	29.22
			7	21.54	26.31	4	24.08	29.31
0.9729	**18.92**	**23.19**	6	21.62	26.40	3	24.15	29.40
8	19.00	23.28	5	21.69	26.49	2	24.23	29.49
7	19.08	23.38	4	21.77	26.58	1	24.31	29.58
6	19.17	23.48	3	21.85	26.67	0	24.38	29.67
5	19.25	23.58	2	21.92	26.77			
4	19.33	23.68	1	22.00	26.86	**0.9659**	**24.46**	**29.76**
3	19.42	23.78	0	22.08	26.95	8	24.54	29.86
2	19.50	23.88				7	24.62	29.95
1	19.58	23.98	**0.9689**	**22.15**	**27.04**	6	24.69	30.04
0	19.67	24.08	8	22.23	27.13	5	24.77	30.13
			7	22.31	27.22	4	24.85	30.22
0.9719	**19.75**	**24.18**	6	22.38	27.31	3	24.92	30.31
8	19.83	24.28	5	22.46	27.40	2	25.00	30.40
7	19.92	24.38	4	22.54	27.49			

TABLE XI

EXTRACT IN WINE

Per cent by Weight

(According to Windisch)

Sp. gr.	Extract.	Sp. gr.	Extract.	Sp. gr.	Extract.	Sp. gr.	Extract.	Sp. gr.	Extract.	Sp. gr.	Extract.
1.0000	0.00	1.0200	5.17	1.0400	10.35	1.0600	15.55	1.0800	20.78	1.1000	26.04
1.0005	0.13	1.0205	5.30	1.0405	10.48	1.0605	15.68	1.0805	20.91	1.1005	26.17
1.0010	0.26	1.0210	5.43	1.0410	10.61	1.0610	15.81	1.0810	21.04	1.1010	26.30
1.0015	0.39	1.0215	5.56	1.0415	10.74	1.0615	15.94	1.0815	21.17	1.1015	26.43
1.0020	0.52	1.0220	5.69	1.0420	10.87	1.0620	16.07	1.0820	21.31	1.1020	26.56
1.0025	0.64	1.0225	5.82	1.0425	11.00	1.0625	16.21	1.0825	21.44	1.1025	26.70
1.0030	0.77	1.0230	5.94	1.0430	11.13	1.0630	16.33	1.0830	21.57	1.1030	26.83
1.0035	0.90	1.0235	6.07	1.0435	11.26	1.0635	16.47	1.0835	21.70	1.1035	26.96
1.0040	1.03	1.0240	6.20	1.0440	11.39	1.0640	16.60	1.0840	21.83	1.1040	27.09
1.0045	1.16	1.0245	6.33	1.0445	11.52	1.0645	16.73	1.0845	21.96	1.1045	27.22
1.0050	1.29	1.0250	6.46	1.0450	11.65	1.0650	16.86	1.0850	22.09	1.1050	27.35
1.0055	1.42	1.0255	6.59	1.0455	11.78	1.0655	16.99	1.0855	22.22	1.1055	27.49
1.0060	1.55	1.0260	6.72	1.0460	11.91	1.0660	17.12	1.0860	22.36	1.1060	27.62
1.0065	1.68	1.0265	6.85	1.0465	12.04	1.0665	17.25	1.0865	22.49	1.1065	27.75
1.0070	1.81	1.0270	6.98	1.0470	12.17	1.0670	17.38	1.0870	22.62	1.1070	27.88
1.0075	1.94	1.0275	7.11	1.0475	12.30	1.0675	17.51	1.0875	22.75	1.1075	28.01
1.0080	2.07	1.0280	7.24	1.0480	12.43	1.0680	17.64	1.0880	22.88	1.1080	28.15
1.0085	2.19	1.0285	7.37	1.0485	12.56	1.0685	17.77	1.0885	23.01	1.1085	28.28
1.0090	2.32	1.0290	7.50	1.0490	12.69	1.0690	17.90	1.0890	23.14	1.1090	28.41
1.0095	2.45	1.0295	7.63	1.0495	12.82	1.0695	18.03	1.0895	23.28	1.1095	28.54
1.0100	2.58	1.0300	7.76	1.0500	12.95	1.0700	18.16	1.0900	23.41	1.1100	28.67
1.0105	2.71	1.0305	7.89	1.0505	13.08	1.0705	18.30	1.0905	23.54	1.1105	28.81
1.0110	2.84	1.0310	8.02	1.0510	13.21	1.0710	18.43	1.0910	23.67	1.1110	28.94
1.0115	2.97	1.0315	8.14	1.0515	13.34	1.0715	18.56	1.0915	23.80	1.1115	29.07
1.0120	3.10	1.0320	8.27	1.0520	13.47	1.0720	18.69	1.0920	23.93	1.1120	29.20
1.0125	3.23	1.0325	8.40	1.0525	13.60	1.0725	18.82	1.0925	24.07	1.1125	29.33
1.0130	3.36	1.0330	8.53	1.0530	13.73	1.0730	18.95	1.0930	24.20	1.1130	29.47
1.0135	3.49	1.0335	8.66	1.0535	13.86	1.0735	19.08	1.0935	24.33	1.1135	29.60
1.0140	3.62	1.0340	8.79	1.0540	13.99	1.0740	19.21	1.0940	24.46	1.1140	29.73
1.0145	3.75	1.0345	8.92	1.0545	14.12	1.0745	19.34	1.0945	24.59	1.1145	29.86
1.0150	3.87	1.0350	9.05	1.0550	14.25	1.0750	19.47	1.0950	24.72	1.1150	29.99
1.0155	4.00	1.0355	9.18	1.0555	14.38	1.0755	19.60	1.0955	24.85	1.1155	30.13
1.0160	4.13	1.0360	9.31	1.0560	14.51	1.0760	19.73	1.0960	24.99		
1.0165	4.26	1.0365	9.44	1.0565	14.64	1.0765	19.86	1.0965	25.12		
1.0170	4.39	1.0370	9.57	1.0570	14.77	1.0770	20.00	1.0970	25.25		
1.0175	4.52	1.0375	9.70	1.0575	14.90	1.0775	20.12	1.0975	25.38		
1.0180	4.65	1.0380	9.83	1.0580	15.03	1.0780	20.26	1.0980	25.51		
1.0185	4.78	1.0385	9.96	1.0585	15.16	1.0785	20.39	1.0985	25.64		
1.0190	4.91	1.0390	10.09	1.0590	15.29	1.0790	20.52	1.0990	25.78		
1.0195	5.04	1.0395	10.22	1.0595	15.42	1.0795	20.65	1.0995	25.91		

TABLE XII

TABLE FOR REDUCING SUGAR CONDENSED FROM THAT OF MUNSON AND WALKER

(Expressed in milligrams)

Cuprous oxide. (Cu_2O).	Dextrose.	Invert sugar.	Lactose. $C_{12}H_{22}O_{11}+H_2O$.	Maltose. $C_{12}H_{22}O_{11}$.	Cuprous oxide. (Cu_2O).	Dextrose.	Invert sugar.	Lactose. $C_{12}H_{22}O_{11}+H_2O$.	Maltose. $C_{12}H_{22}O_{11}$.
10	4.0	4.5	4.0	5.9	260	117.6	121.4	178.3	203.9
15	6.2	6.7	7.5	9.9	265	120.0	123.9	181.9	207.9
20	8.3	8.9	10.9	13.8	270	122.5	126.4	185.4	211.8
25	10.5	11.2	14.4	17.8	275	124.9	128.9	188.9	215.8
30	12.6	13.4	17.8	21.8	280	127.3	131.4	192.4	219.7
35	14.8	15.6	21.3	25.7	285	129 8	133.9	196 0	223.7
40	16.9	17.8	24.8	29.7	290	132.3	136.4	199.5	227.6
45	19.1	20.1	28.2	33.7	295	134.7	138.9	203.0	231.6
50	21.3	22.3	31.7	37.6	300	137.2	141.5	206 6	235.5
55	23.5	24.6	35.1	41.6	305	139.7	144.0	210.1	239.5
60	25.6	26.8	38.6	45.6	310	142.2	146.6	213.7	243.5
65	27.8	29.1	42.1	49.5	315	144.7	149.1	217.2	247.4
70	30.0	31.3	45.5	53.5	320	147.2	151.7	220.7	251.3
75	32.2	33.6	49.0	57.5	325	149.7	154.3	224.3	255 3
80	34.4	35.9	52.5	61.4	330	152.2	156.8	227.8	259.3
85	36.7	38.2	56.0	65.4	335	154.7	159.4	231.4	263.2
90	38.9	40.4	59.4	69.3	340	157.3	162.0	234.9	267.1
95	41.1	42.7	62.9	73.3	345	159.8	164.6	238.5	271.1
100	43.3	45.0	66.4	77.3	350	162.4	167.2	242.0	275.0
105	45.5	47.3	69.8	81.2	355	164.9	169.8	245.6	279.0
110	47.8	49.6	73.3	85.2	360	167.5	172.5	249.1	282.9
115	50.0	51.9	76.8	89.2	365	170.1	175.1	252.7	286.9
120	52.3	54.3	80.3	93.1	370	172.7	177 7	256.2	290.8
125	54.5	56.6	83.8	97.1	375	175.3	180.4	259.8	294.8
130	56.8	58.9	87.3	101.0	380	177.9	183.0	263.4	298.7
135	59.0	61.2	90.8	105.0	385	180.5	185 7	266 9	302 7
140	61.3	63.6	94.2	109.0	390	183.1	188.4	270.5	366 6
145	63.6	65.9	97.7	112.9	395	185.7	191.0	274 0	310 6
150	65.9	68.3	101.2	116.9	400	188.4	193.7	277 6	314.5
155	68.2	70.6	104.7	120.8	405	191.0	196.4	281 1	318.5
160	70.4	73.0	108.2	124.8	410	193.7	199.1	284.7	322.4
165	72.8	75.3	111.7	128.8	415	196.3	201.8	288.3	326.3
170	75.1	77.7	115.2	132.7	420	199.0	204.6	291.9	330.3
175	77.4	80.1	118.7	136.7	425	201.7	207.3	295.4	334.2
180	79.7	82.5	122.2	140.6	430	204.4	210.0	299.0	338.2
185	84.2	84.9	125.7	144.6	435	207.1	212.8	302.6	342.1
190	84.3	87.2	129.2	148.6	440	209.8	215.5	306.2	346.1
195	86.7	89.6	132.7	152.5	445	212.5	218.3	309.7	350.0
200	89.0	92.0	136.2	156.5	450	215.2	221.1	313.3	353 9
205	91.4	94.5	139.7	160.4	455	218.0	223.9	316 9	357 9
210	93.7	96.9	143.2	164.4	460	220.7	226.7	320.5	361.8
215	96.1	99.3	146.7	168.3	465	223.5	229.5	324.1	365.8
220	98.4	101.7	150.2	172.3	470	226.2	232.3	327.7	369.7
225	100.8	104.2	153.7	176.2	475	229.0	235.1	331.3	373.7
230	103.2	106.6	157.2	180.2	480	231.8	237.9	334.8	377.6
235	105.6	109.1	160.7	184.2	485	234.6	240.8	338.4	381.5
240	108.0	111.5	164.3	188.1	490	237.4	243.6	342.0	385.5
245	110.4	114 0	167.8	192.1					
250	112.8	116 4	171.3	196.0					
255	115.2	118.9	174.8	200.0					

TABLE XIII

EXTRACT IN BEER-WORT

(According to Schultz and Ostermann)

Specific gravity at 15° C.	Extract. Per cent by weight.	Specific gravity at 15° C.	Extract. Per cent by weight.	Specific gravity at 15° C.	Extract. Per cent by weight.	Specific gravity at 15° C.	Extract. Per cent by weight.
1.0000	0.00	1.0235	6.07	1.0470	11.89	1.0705	17.59
1.0005	0.13	1.0240	6.19	1.0475	12.01	1.0710	17.70
1.0010	0.26	1.0245	6.31	1.0480	12.14	1.0715	17.81
1.0015	0.39	1.0250	6.44	1.0485	12.26	1.0720	17.93
1.0020	0.52	1.0255	6.58	1.0490	12.38	1.0725	18.04
1.0025	0.66	1.0260	6.71	1.0495	12.50	1.0730	18.15
1.0030	0.79	1.0265	6.85	1.0500	12.63	1.0735	18.26
1.0035	0.92	1.0270	6.99	1.0505	12.75	1.0740	18.38
1.0040	1.05	1.0275	7.12	1.0510	12.87	1.0745	18.49
1.0045	1.18	1.0280	7.26	1.0515	12.99	1.0750	18.59
1.0050	1.31	1.0285	7.37	1.0520	13.12	1.0755	18.70
1.0055	1.44	1.0290	7.48	1.0525	13.24	1.0760	18.81
1.0060	1.56	1.0295	7.60	1.0530	13.36	1.0765	18.91
1.0065	1.69	1.0300	7.71	1.0535	13.48	1.0770	19.02
1.0070	1.82	1.0305	7.82	1.0540	13.61	1.0775	19.12
1.0075	1.95	1.0310	7.93	1.0545	13.73	1.0780	19.23
1.0080	2.07	1.0315	8.04	1.0550	13.86	1.0785	19.33
1.0085	2.20	1.0320	8.16	1.0555	13.98	1.0790	19.44
1.0090	2.33	1.0325	8.27	1.0560	14.11	1.0795	19.56
1.0095	2.46	1.0330	8.40	1.0565	14.23	1.0800	19.67
1.0100	2.58	1.0335	8.53	1.0570	14.36	1.0805	19.79
1.0105	2.71	1.0340	8.67	1.0575	14.49	1.0810	19.91
1.0110	2.84	1.0345	8.80	1.0580	14.62	1.0815	20.03
1.0115	2.97	1.0350	8.94	1.0585	14.75	1.0820	20.14
1.0120	3.10	1.0355	9.07	1.0590	14.89	1.0825	20.26
1.0125	3.23	1.0360	9.21	1.0595	15.02	1.0830	20.37
1.0130	3.35	1.0365	9.34	1.0600	15.14	1.0835	20.48
1.0135	3.48	1.0370	9.45	1.0605	15.25	1.0840	20.59
1.0140	3.61	1.0375	9.57	1.0610	15.36	1.0845	20.70
1.0145	3.74	1.0380	9.69	1.0615	15.47	1.0850	20.81
1.0150	3.87	1.0385	9.81	1.0620	15.58	1.0855	20.93
1.0155	4.00	1.0390	9.92	1.0625	15.69	1.0860	21.06
1.0160	4.13	1.0395	10.04	1.0630	15.80	1.0865	21.19
1.0165	4.26	1.0400	10.16	1.0635	15.92	1.0870	21.33
1.0170	4.39	1.0405	10.27	1.0640	16.03	1.0875	21.43
1.0175	4.53	1.0410	10.40	1.0645	16.14	1.0880	21.54
1.0180	4.66	1.0415	10.52	1.0650	16.25	1.0885	21.64
1.0185	4.79	1.0420	10.65	1.0655	16.37	1.0890	21.75
1.0190	4.93	1.0425	10.77	1.0660	16.50	1.0895	21.86
1.0195	5.06	1.0430	10.90	1.0665	16.62	1.0900	21.98
1.0200	5.20	1.0435	11.03	1.0670	16.74	1.0905	22.08
1.0205	5.33	1.0440	11.15	1.0675	16.86	1.0910	22.19
1.0210	5.45	1.0445	11.28	1.0680	16.99	1.0915	22.30
1.0215	5.57	1.0450	11.40	1.0685	17.11	1.0920	22.41
1.0220	5.70	1.0455	11.53	1.0690	17.23	1.0925	22.52
1.0225	5.82	1.0460	11.65	1.0695	17.35	1.0930	22.63
1.0230	5.94	1.0465	11.77	1.0700	17.48	1.0935	22.73

TABLE XIII. — (*Continued*)

EXTRACT IN BEER-WORT

(According to Schultz and Ostermann)

Specific gravity at 15° C.	Extract. Per cent by weight.	Specific gravity at 15° C.	Extract. Per cent by weight.	Specific gravity at 15° C.	Extract. Per cent by weight.	Specific gravity at 15° C.	Extract. Per cent by weight.
1.0940	22.84	1.1020	24.53	1.1100	26.27	1.1180	27.88
1.0945	22.94	1.1025	24.64	1.1105	26.37	1.1185	27.98
1.0950	23.05	1.1030	24.74	1.1110	26.48	1.1190	28.09
1.0955	23.16	1.1035	24.85	1.1115	26.58	1.1195	28.19
1.0960	23.27	1.1040	24.96	1.1120	26.68	1.1200	28.28
1.0965	23.37	1.1045	25.07	1.1125	26.79	1.1205	28.38
1.0970	23.48	1.1050	25.18	1.1130	26.89	1.1210	28.48
1.0975	23.59	1.1055	25.29	1.1135	26.99	1.1215	28.58
1.0980	23.69	1.1060	25.40	1.1140	27.09	1.1220	28.68
1.0985	23.80	1.1065	25.50	1.1145	27.19	1.1225	28.78
1.0990	23.90	1.1070	25.61	1.1150	27.29	1.1230	28.88
1.0995	24.01	1.1075	25.71	1.1155	27.38	1.1235	28.98
1.1000	24.11	1.1080	25.82	1.1160	27.48	1.1240	29.08
1.1005	24.21	1.1085	25.93	1.1165	27.58	1.1245	29.18
1.1010	24.32	1.1090	26.05	1.1170	27.68	1.1250	29.28
1.1015	24.43	1.1095	26.16	1.1175	27.78	1.1255	29.38

LOGARITHMS OF NUMBERS

Natural numbers.	0	1	2	3	4	5	6	7	8	9	Proportional parts.							
											1	2	3	4	5	6	7	8	9
10	0000	0043	0086	0128	0170	0212	0253	0294	0334	0374	4	8	12	17	21	25	29	33	37
11	0414	0453	0492	0531	0569	0607	0645	0682	0719	0755	4	8	11	15	19	23	26	30	34
12	0792	0828	0864	0899	0934	0969	1004	1038	1072	1106	3	7	10	14	17	21	24	28	31
13	1139	1173	1206	1239	1271	1303	1335	1367	1399	1430	3	6	10	13	16	19	23	26	29
14	1461	1492	1523	1553	1584	1614	1644	1673	1703	1732	3	6	9	12	15	18	21	24	27
15	1761	1790	1818	1847	1875	1903	1931	1959	1987	2014	3	6	8	11	14	17	20	22	25
16	2041	2068	2095	2122	2148	2175	2201	2227	2253	2279	3	5	8	11	13	16	18	21	24
17	2304	2330	2355	2380	2405	2430	2455	2480	2504	2529	2	5	7	10	12	15	17	20	22
18	2553	2577	2601	2625	2648	2672	2695	2718	2742	2765	2	5	7	9	12	14	16	19	21
19	2788	2810	2833	2856	2878	2900	2923	2945	2967	2989	2	4	7	9	11	13	16	18	20
20	3010	3032	3054	3075	3096	3118	3139	3160	3181	3201	2	4	6	8	11	13	15	17	19
21	3222	3243	3263	3284	3304	3324	3345	3365	3385	3404	2	4	6	8	10	12	14	16	18
22	3424	3444	3464	3483	3502	3522	3541	3560	3579	3598	2	4	6	8	10	12	14	15	17
23	3617	3636	3655	3674	3692	3711	3729	3747	3766	3784	2	4	6	7	9	11	13	15	17
24	3802	3820	3838	3856	3874	3892	3909	3927	3945	3962	2	4	5	7	9	11	12	14	16
25	3979	3997	4014	4031	4048	4065	4082	4099	4116	4133	2	3	5	7	9	10	12	14	15
26	4150	4166	4183	4200	4216	4232	4249	4265	4281	4298	2	3	5	7	8	10	11	13	15
27	4314	4330	4346	4362	4378	4393	4409	4425	4440	4456	2	3	5	6	8	9	11	13	14
28	4472	4487	4502	4518	4533	4548	4564	4579	4594	4609	2	3	5	6	8	9	11	12	14
29	4624	4639	4654	4669	4683	4698	4713	4728	4742	4757	1	3	4	6	7	9	10	12	13
30	4771	4786	4800	4814	4829	4843	4857	4871	4886	4900	1	3	4	6	7	9	10	11	13
31	4914	4928	4942	4955	4969	4983	4997	5011	5024	5038	1	3	4	6	7	8	10	11	12
32	5051	5065	5079	5092	5105	5119	5132	5145	5159	5172	1	3	4	5	7	8	9	11	12
33	5185	5198	5211	5224	5237	5250	5263	5276	5289	5302	1	3	4	5	6	8	9	10	12
34	5315	5328	5340	5353	5366	5378	5391	5403	5416	5428	1	3	4	5	6	8	9	10	11
35	5441	5453	5465	5478	5490	5502	5514	5527	5539	5551	1	2	4	5	6	7	9	10	11
36	5563	5575	5587	5599	5611	5623	5635	5647	5658	5670	1	2	4	5	6	7	8	10	11
37	5682	5694	5705	5717	5729	5740	5752	5763	5775	5786	1	2	3	5	6	7	8	9	10
38	5798	5809	5821	5832	5843	5855	5866	5877	5888	5899	1	2	3	5	6	7	8	9	10
39	5911	5922	5933	5944	5955	5966	5977	5988	5999	6010	1	2	3	4	5	7	8	9	10
40	6021	6031	6042	6053	6064	6075	6085	6096	6107	6117	1	2	3	4	5	6	8	9	10
41	6128	6138	6149	6160	6170	6180	6191	6201	6212	6222	1	2	3	4	5	6	7	8	9
42	6232	6243	6253	6263	6274	6284	6294	6304	6314	6325	1	2	3	4	5	6	7	8	9
43	6335	6345	6355	6365	6375	6385	6395	6405	6415	6425	1	2	3	4	5	6	7	8	9
44	6435	6444	6454	6464	6474	6484	6493	6503	6513	6522	1	2	3	4	5	6	7	8	9
45	6532	6542	6551	6561	6571	6580	6590	6599	6609	6618	1	2	3	4	5	6	7	8	9
46	6628	6637	6646	6656	6665	6675	6684	6693	6702	6712	1	2	3	4	5	6	7	7	8
47	6721	6730	6739	6749	6758	6767	6776	6785	6794	6803	1	2	3	4	5	5	6	7	8
48	6812	6821	6830	6839	6848	6857	6866	6875	6884	6893	1	2	3	4	4	5	6	7	8
49	6902	6911	6920	6928	6937	6946	6955	6964	6972	6981	1	2	3	4	4	5	6	7	8
50	6990	6998	7007	7016	7024	7033	7042	7050	7059	7067	1	2	3	3	4	5	6	7	8
51	7076	7084	7093	7101	7110	7118	7126	7135	7143	7152	1	2	3	3	4	5	6	7	8
52	7160	7168	7177	7185	7193	7202	7210	7218	7226	7235	1	2	2	3	4	5	6	7	7
53	7243	7251	7259	7267	7275	7284	7292	7300	7308	7316	1	2	2	3	4	5	6	6	7
54	7324	7332	7340	7348	7356	7364	7372	7380	7388	7396	1	2	2	3	4	5	6	6	7

LOGARITHMS OF NUMBERS

Natural numbers.	0	1	2	3	4	5	6	7	8	9	Proportional parts.								
											1	2	3	4	5	6	7	8	9
55	7404	7412	7419	7427	7435	7443	7451	7459	7466	7474	1	2	2	3	4	5	5	6	7
56	7482	7490	7497	7505	7513	7520	7528	7536	7543	7551	1	2	2	3	4	5	5	6	7
57	7559	7566	7574	7582	7589	7597	7604	7612	7619	7627	1	2	2	3	4	5	5	6	7
58	7634	7642	7649	7657	7664	7672	7679	7686	7694	7701	1	1	2	3	4	4	5	6	7
59	7709	7716	7723	7731	7738	7745	7752	7760	7767	7774	1	1	2	3	4	4	5	6	7
60	7782	7789	7796	7803	7810	7818	7825	7832	7839	7846		1	2	3	4	4	5	6	6
61	7853	7860	7868	7875	7882	7889	7896	7903	7910	7917	1	1	2	3	4	4	5	6	6
62	7924	7931	7938	7945	7952	7959	7966	7973	7980	7987	1	1	2	3	3	4	5	6	6
63	7993	8000	8007	8014	8021	8028	8035	8041	8048	8055	1	1	2	3	3	4	5	5	6
64	8062	8069	8075	8082	8089	8096	8102	8109	8116	8122	1	1	2	3	3	4	5	5	6
65	8129	8136	8142	8149	8156	8162	8169	8176	8182	8189	1	1	2	3	3	4	5	5	6
66	8195	8202	8209	8215	8222	8228	8235	8241	8248	8254	1	1	2	3	3	4	5	5	6
67	8261	8267	8274	8280	8287	8293	8299	8306	8312	8319	1	1	2	3	3	4	5	5	6
68	8325	8331	8338	8344	8351	8357	8363	8370	8376	8382	1	1	2	3	3	4	4	5	6
69	8388	8395	8401	8407	8414	8420	8426	8432	8439	8445	1	1	2	2	3	4	4	5	6
70	8451	8457	8463	8470	8476	8482	8488	8494	8500	8506	1	1	2	2	3	4	4	5	6
71	8513	8519	8525	8531	8537	8543	8549	8555	8561	8567	1	1	2	2	3	4	4	5	5
72	8573	8579	8585	8591	8597	8603	8609	8615	8621	8627	1	1	2	2	3	4	4	5	5
73	8633	8639	8645	8651	8657	8663	8669	8675	8681	8686	1	1	2	2	3	4	4	5	5
74	8692	8698	8704	8710	8716	8722	8727	8733	8739	8745	1	1	2	2	3	4	4	5	5
75	8751	8756	8762	8768	8774	8779	8785	8791	8797	8802	1	1	2	2	3	3	4	5	5
76	8808	8814	8820	8825	8831	8837	8842	8848	8854	8859	1	1	2	2	3	3	4	5	5
77	8865	8871	8876	8882	8887	8893	8899	8904	8910	8915	1	1	2	2	3	3	4	4	5
78	8921	8927	8932	8938	8943	8949	8954	8960	8965	8971	1	1	2	2	3	3	4	4	5
79	8976	8982	8987	8993	8998	9004	9009	9015	9020	9026	1	1	2	2	3	3	4	4	5
80	9031	9036	9042	9047	9053	9058	9063	9069	9074	9079	1	1	2	2	3	3	4	4	5
81	9085	9090	9096	9101	9106	9112	9117	9122	9128	9133	1	1	2	2	3	3	4	4	5
82	9138	9143	9149	9154	9159	9165	9170	9175	9180	9186	1	1	2	2	3	3	4	4	5
83	9191	9196	9201	9206	9212	9217	9222	9227	9232	9238	1	1	2	2	3	3	4	4	5
84	9243	9248	9253	9258	9263	9269	9274	9279	9284	9289	1	1	2	2	3	3	4	4	5
85	9294	9299	9304	9309	9315	9320	9325	9330	9335	9340	1	1	2	2	3	3	4	4	5
86	9345	9350	9355	9360	9365	9370	9375	9380	9385	9390	1	1	2	2	3	3	4	4	5
87	9395	9400	9405	9410	9415	9420	9425	9430	9435	9440	0	1	1	2	2	3	3	4	4
88	9445	9450	9455	9460	9465	9469	9474	9479	9484	9489	0	1	1	2	2	3	3	4	4
89	9494	9499	9504	9509	9513	9518	9523	9528	9533	9538	0	1	1	2	2	3	3	4	4
90	9542	9547	9552	9557	9562	9566	9571	9576	9581	9586	0	1	1	2	2	3	3	4	4
91	9590	9595	9600	9605	9609	9614	9619	9624	9628	9633	0	1	1	2	2	3	3	4	4
92	9638	9643	9647	9652	9657	9661	9666	9671	9675	9680	0	1	1	2	2	3	3	4	4
93	9685	9689	9694	9699	9703	9708	9713	9717	9722	9727	0	1	1	2	2	3	3	4	4
94	9731	9736	9741	9745	9750	9754	9759	9763	9768	9773	0	1	1	2	2	3	3	4	4
95	9777	9782	9786	9791	9795	9800	9805	9800	9814	9818	0	1	1	2	2	3	3	4	4
96	9823	9827	9832	9836	9841	9845	9850	9854	9859	9863	0	1	1	2	2	3	3	4	4
97	9868	9872	9877	9881	9886	9890	9894	9899	9903	9908	0	1	1	2	2	3	3	4	4
98	9912	9917	9921	9926	9930	9934	9939	9943	9948	9952	0	1	1	2	2	3	3	4	4
99	9956	9961	9965	9969	9974	9978	9983	9987	9991	9996	0	1	1	2	2	3	3	3	4

ANTILOGARITHMS

Logarithms.	0	1	2	3	4	5	6	7	8	9	Proportional parts.								
											1	2	3	4	5	6	7	8	9
0.00	1000	1002	1005	1007	1009	1012	1014	1016	1019	1021	0	0	1	1	1	1	2	2	2
0.01	1023	1026	1028	1030	1033	1035	1038	1040	1042	1045	0	0	1	1	1	1	2	2	2
0.02	1047	1050	1052	1054	1057	1059	1062	1064	1067	1069	0	0	1	1	1	1	2	2	2
0.03	1072	1074	1076	1079	1081	1084	1086	1089	1091	1094	0	0	1	1	1	1	2	2	2
0.04	1096	1099	1102	1104	1107	1109	1112	1114	1117	1119	0	1	1	1	1	2	2	2	2
0.05	1122	1125	1127	1130	1132	1135	1138	1140	1143	1146	0	1	1	1	1	2	2	2	2
0.06	1148	1151	1153	1156	1159	1161	1164	1167	1169	1172	0	1	1	1	1	2	2	2	2
0.07	1175	1178	1180	1183	1186	1189	1191	1194	1197	1199	0	1	1	1	1	2	2	2	2
0.08	1202	1205	1208	1211	1213	1216	1219	1222	1225	1227	0	1	1	1	1	2	2	2	3
0.09	1230	1233	1236	1239	1242	1245	1247	1250	1253	1256	0	1	1	1	1	2	2	2	3
0.10	1259	1262	1265	1268	1271	1274	1276	1279	1282	1285	0	1	1	1	1	2	2	2	3
0.11	1288	1291	1294	1297	1300	1303	1306	1309	1312	1315	0	1	1	1	2	2	2	2	3
0.12	1318	1321	1324	1327	1330	1334	1337	1340	1343	1346	0	1	1	1	2	2	2	2	3
0.13	1349	1352	1355	1358	1361	1365	1368	1371	1374	1377	0	1	1	1	2	2	2	3	3
0.14	1380	1384	1387	1390	1393	1396	1400	1403	1406	1409	0	1	1	1	2	2	2	3	3
0.15	1413	1416	1419	1422	1426	1429	1432	1435	1439	1442	0	1	1	1	2	2	2	3	3
0.16	1445	1449	1452	1455	1459	1462	1466	1469	1472	1476	0	1	1	1	2	2	2	3	3
0.17	1479	1483	1486	1489	1493	1496	1500	1503	1507	1510	0	1	1	1	2	2	2	3	3
0.18	1514	1517	1521	1524	1528	1531	1535	1538	1542	1545	0	1	1	1	2	2	2	3	3
0.19	1549	1552	1556	1560	1563	1567	1570	1574	1578	1581	0	1	1	1	2	2	3	3	3
0.20	1585	1589	1592	1596	1600	1603	1607	1611	1614	1618	0	1	1	1	2	2	3	3	3
0.21	1622	1626	1629	1633	1637	1641	1644	1648	1652	1656	0	1	1	2	2	2	3	3	3
0.22	1660	1663	1667	1671	1675	1679	1683	1687	1690	1694	0	1	1	2	2	2	3	3	3
0.23	1698	1702	1706	1710	1714	1718	1722	1726	1730	1734	0	1	1	2	2	2	3	3	4
0.24	1738	1742	1746	1750	1754	1758	1762	1766	1770	1774	0	1	1	2	2	2	3	3	4
0.25	1778	1782	1786	1791	1795	1799	1803	1807	1811	1816	0	1	1	2	2	2	3	3	4
0.26	1820	1824	1828	1832	1837	1841	1845	1849	1854	1858	0	1	1	2	2	3	3	3	4
0.27	1862	1866	1871	1875	1879	1884	1888	1892	1897	1901	0	1	1	2	2	3	3	3	4
0.28	1905	1910	1914	1919	1923	1928	1932	1936	1941	1945	0	1	1	2	2	3	3	4	4
0.29	1950	1954	1959	1963	1968	1972	1977	1982	1986	1991	0	1	1	2	2	3	3	4	4
0.30	1995	2000	2004	2009	2014	2018	2023	2028	2032	2037	0	1	1	2	2	3	3	4	4
0.31	2042	2046	2051	2056	2061	2065	2070	2075	2080	2084	0	1	1	2	2	3	3	4	4
0.32	2089	2094	2099	2104	2109	2113	2118	2123	2128	2133	0	1	1	2	2	3	3	4	4
0.33	2138	2143	2148	2153	2158	2163	2168	2173	2178	2183	0	1	1	2	2	3	3	4	4
0.34	2188	2193	2198	2203	2208	2213	2218	2223	2228	2234	1	1	2	2	3	3	4	4	5
0.35	2239	2244	2249	2254	2259	2265	2270	2275	2280	2286	1	1	2	2	3	3	4	4	5
0.36	2291	2296	2301	2307	2312	2317	2323	2328	2333	2339	1	1	2	2	3	3	4	4	5
0.37	2344	2350	2355	2360	2366	2371	2377	2382	2388	2393	1	1	2	2	3	3	4	4	5
0.38	2399	2404	2410	2415	2421	2427	2432	2438	2443	2449	1	1	2	2	3	3	4	4	5
0.39	2455	2460	2466	2472	2477	2483	2489	2495	2500	2506	1	1	2	2	3	3	4	5	5
0.40	2512	2518	2523	2529	2535	2541	2547	2553	2559	2564	1	1	2	2	3	4	4	5	5
0.41	2570	2576	2582	2588	2594	2600	2606	2612	2618	2624	1	1	2	2	3	4	4	5	5
0.42	2630	2636	2642	2649	2655	2661	2667	2673	2679	2685	1	1	2	2	3	4	4	5	6
0.43	2692	2698	2704	2710	2716	2723	2729	2735	2742	2748	1	1	2	3	3	4	4	5	6
0.44	2754	2761	2767	2773	2780	2786	2793	2799	2805	2812	1	1	2	3	3	4	4	5	6
0.45	2818	2825	2831	2838	2844	2851	2858	2864	2871	2877	1	1	2	3	3	4	5	5	6
0.46	2884	2891	2897	2904	2911	2917	2924	2931	2938	2944	1	1	2	3	3	4	5	5	6
0.47	2951	2958	2965	2972	2979	2985	2992	2999	3006	3013	1	1	2	3	3	4	5	5	6
0.48	3020	3027	3034	3041	3048	3055	3062	3069	3076	3083	1	1	2	3	4	4	5	6	6
0.49	3090	3097	3105	3112	3119	3126	3133	3141	3148	3155	1	1	2	3	4	4	5	6	6

ANTILOGARITHMS

Logarithms.	0	1	2	3	4	5	6	7	8	9	Proportional parts.							
											1	2	3	4	5	6	7	8	9
0.50	3162	3170	3177	3184	3192	3199	3206	3214	3221	3228	1	1	2	3	4	4	5	6	7
0.51	3236	3243	3251	3258	3266	3273	3281	3289	3296	3304	1	2	2	3	4	5	5	6	7
0.52	3311	3319	3327	3334	3342	3350	3357	3365	3373	3381	1	2	2	3	4	5	5	6	7
0.53	3388	3396	3404	3412	3420	3428	3436	3443	3451	3459	1	2	2	3	4	5	6	6	7
0.54	3467	3475	3483	3491	3499	3508	3516	3524	3532	3540	1	2	2	3	4	5	6	6	7
0.55	3548	3556	3565	3573	3581	3589	3597	3606	3614	3622	1	2	2	3	4	5	6	7	7
0.56	3631	3639	3648	3656	3664	3673	3681	3690	3698	3707	1	2	3	3	4	5	6	7	8
0.57	3715	3724	3733	3741	3750	3758	3767	3776	3784	3793	1	2	3	3	4	5	6	7	8
0.58	3802	3811	3819	3828	3837	3846	3855	3864	3873	3882	1	2	3	4	4	5	6	7	8
0.59	3890	3899	3908	3917	3926	3936	3945	3954	3963	3972	1	2	3	4	5	5	6	7	8
0.60	3981	3990	3999	4009	4018	4027	4036	4046	4055	4064	1	2	3	4	5	6	6	7	8
0.61	4074	4083	4093	4102	4111	4121	4130	4140	4150	4159	1	2	3	4	5	6	7	8	9
0.62	4169	4178	4188	4198	4207	4217	4227	4236	4246	4256	1	2	3	4	5	6	7	8	9
0.63	4266	4276	4285	4295	4305	4315	4325	4335	4345	4355	1	2	3	4	5	6	7	8	9
0.64	4365	4375	4385	4395	4406	4416	4426	4436	4446	4457	1	2	3	4	5	6	7	8	9
0.65	4467	4477	4487	4498	4508	4519	4529	4539	4550	4560	1	2	3	4	5	6	7	8	9
0.66	4571	4581	4592	4603	4613	4624	4634	4645	4656	4667	1	2	3	4	5	6	7	9	10
0.67	4677	4688	4699	4710	4721	4732	4742	4753	4764	4775	1	2	3	4	5	7	8	9	10
0.68	4786	4797	4808	4819	4831	4842	4853	4864	4875	4887	1	2	3	4	6	7	8	9	10
0.69	4898	4909	4920	4932	4943	4955	4966	4977	4989	5000	1	2	3	5	6	7	8	9	10
0.70	5012	5023	5035	5047	5058	5070	5082	5093	5105	5117	1	2	4	5	6	7	8	9	11
0.71	5129	5140	5152	5164	5176	5188	5200	5212	5224	5236	1	2	4	5	6	7	8	10	11
0.72	5248	5260	5272	5284	5297	5309	5321	5333	5346	5358	1	2	4	5	6	7	9	10	11
0.73	5370	5383	5395	5408	5420	5433	5445	5458	5470	5483	1	3	4	5	6	8	9	10	11
0.74	5495	5508	5521	5534	5546	5559	5572	5585	5598	5610	1	3	4	5	6	8	9	10	12
0.75	5623	5636	5649	5662	5675	5689	5702	5715	5728	5741	1	3	4	5	7	8	9	10	12
0.76	5754	5768	5781	5794	5808	5821	5834	5848	5861	5875	1	3	4	5	7	8	9	11	12
0.77	5888	5902	5916	5929	5943	5957	5970	5984	5998	6012	1	3	4	5	7	8	10	11	12
0.78	6026	6039	6053	6067	6081	6095	6109	6124	6138	6152	1	3	4	6	7	8	10	11	13
0.79	6166	6180	6194	6209	6223	6237	6252	6266	6281	6295	1	3	4	6	7	9	10	11	13
0.80	6310	6324	6339	6353	6368	6383	6397	6412	6427	6442	1	3	4	6	7	9	10	12	13
0.81	6457	6471	6486	6501	6516	6531	6546	6561	6577	6592	2	3	5	6	8	9	11	12	14
0.82	6607	6622	6637	6653	6668	6683	6699	6714	6730	6745	2	3	5	6	8	9	11	12	14
0.83	6761	6776	6792	6808	6823	6839	6855	6871	6887	6902	2	3	5	6	8	9	11	13	14
0.84	6918	6934	6950	6966	6982	6998	7015	7031	7047	7063	2	3	5	6	8	10	11	13	15
0.85	7079	7096	7112	7129	7145	7161	7178	7194	7211	7228	2	3	5	7	8	10	12	13	15
0.86	7244	7261	7278	7295	7311	7328	7345	7362	7379	7396	2	3	5	7	8	10	12	13	15
0.87	7413	7430	7447	7464	7482	7499	7516	7534	7551	7568	2	3	5	7	9	10	12	14	16
0.88	7586	7603	7621	7638	7656	7674	7691	7709	7727	7745	2	4	5	7	9	11	12	14	16
0.89	7762	7780	7798	7816	7834	7852	7870	7889	7907	7925	2	4	5	7	9	11	13	14	16
0.90	7943	7962	7980	7998	8017	8035	8054	8072	8091	8110	2	4	6	7	9	11	13	15	17
0.91	8128	8147	8166	8185	8204	8222	8241	8260	8279	8299	2	4	6	8	9	11	13	15	17
0.92	8318	8337	8356	8375	8395	8414	8433	8453	8472	8492	2	4	6	8	10	12	14	15	17
0.93	8511	8531	8551	8570	8590	8610	8630	8650	8670	8690	2	4	6	8	10	12	14	16	18
0.94	8710	8730	8750	8770	8790	8810	8831	8851	8872	8892	2	4	6	8	10	12	14	16	18
0.95	8913	8933	8954	8974	8995	9016	9036	9057	9078	9099	2	4	6	8	10	12	15	17	19
0.96	9120	9141	9162	9183	9204	9226	9247	9268	9290	9311	2	4	6	8	11	13	15	17	19
0.97	9333	9354	9376	9397	9419	9441	9462	9484	9506	9528	2	4	7	9	11	13	15	17	20
0.98	9550	9572	9594	9616	9638	9661	9683	9705	9727	9750	2	4	7	9	11	13	16	18	20
0.99	9772	9795	9817	9840	9863	9886	9908	9931	9954	9977	2	5	7	9	11	14	16	18	20

APPENDIX B

REAGENTS

Air Analysis

Pettenkofer Method. — *Barium Hydroxide.* — A solution containing about 4 grams of BaO and 0.2 gram of $BaCl_2$ to the liter. (1 c.c. = 1 mg. CO_2, approximately.):

Sulphuric Acid. — Dilute 45.45 c.c. of normal sulphuric acid to one liter. (1 c.c. = 1 mg. CO_2.) To standardize the solution measure 25 c.c. into a weighed platinum dish, add dilute ammonia-water in slight excess, evaporate to dryness on the water-bath, and dry at 120° C. to constant weight.

Standard Lime-water. — (For Popular Tests.) — Shake one part of freshly slaked lime with 20 parts of distilled water for twenty minutes and let the solution stand overnight or until perfectly clear. This solution should be very nearly equivalent to the above standard sulphuric acid. To a liter of distilled water add 2.5 c.c. of a solution of 0.7 gram of phenolphthalein in 100 c.c. of 50 per cent alcohol and add lime-water drop by drop until a slight permanent pink color is produced. Then add 6.3 c.c. of the above calcium hydroxide solution. The resulting solution is the standard lime-water used for the tests.

Water Analysis

For Ammonia. — *Water Free from Ammonia.* — The ammonia-free water used in this laboratory is made by redistilling distilled water from a solution of alkaline permanganate in a steam-heated copper still. Only the middle portion of the distillate is collected. Oftentimes the distillate from a good spring-water may be used.

Nessler's Reagent. — Dissolve 61.750 grams KI in 250 c.c. distilled water and add a cold solution of $HgCl_2$ which has been saturated by boiling with an excess of the salt and allowing it

to crystallize out. Add the $HgCl_2$ cautiously until a slight permanent red precipitate (HgI_2) appears. Dissolve this slight precipitate by adding 0.750 gram powdered KI. Then add 150 grams of KOH dissolved in 250 c.c. of water. Make up to a liter and allow it to stand over night to settle. This solution should give the required color with ammonia within five minutes, and should not precipitate within two hours.

Alkaline Permanganate. — Dissolve 233 grams of the best stick potash in 350 c.c. of distilled water. Filter this strong solution, if necessary, through a layer of glass wool on a porcelain filter-plate. Dilute with 700 to 750 c.c. of distilled water to a specific gravity of 1.125, add 8 grams of potassium permanganate crystals, and boil down to one liter to free the solution from nitrogen. Each new lot of reagent must be tested before being used, but when the chemicals used are all good there should be no correction needed for ammonia in the solution.

Standard Ammonia Solution. — Dissolve 3.82 grams chemically pure NH_4Cl in a liter of water free from ammonia. This is the strong solution from which the standard solution is made by diluting 10 c.c. to a liter with water free from ammonia. One cubic centimeter of the standard solution = 0.00001 gram nitrogen. This solution, like the nitrite standard and other dilute solutions, must be preserved in sterilized bottles protected from dust and organic matter.

For Nitrites. — *Standard Nitrite Solution.* — The pure silver nitrite used in making this solution is prepared by the double decomposition of silver nitrate and potassium nitrite, and repeated crystallizations from water of the rather difficultly soluble silver nitrite. 1.1 grams of this silver nitrite are dissolved in nitrite-free water, the silver completely precipitated by the addition of the standard salt solution used in the determination of chlorine, and the solution made up to 1 liter. 100 c.c. of this strong solution are diluted to 1 liter, and 10 c.c. of this last solution again diluted to 1 liter. The final solution is the one used in preparing standards. 1 c.c. = 0.0000001 gram nitrogen.

Sulphanilic Acid. — Dissolve 3.3 grams sulphanilic acid in

750 c.c. of water by the aid of heat, and add 250 c.c. glacial acetic acid.

Naphthylamine Acetate. — Boil 0.5 gram of α-naphthylamine in 100 c.c. of water in a small Erlenmeyer flask for about five minutes, filter through a plug of washed absorbent cotton, add 250 c.c. glacial acetic acid, and dilute to 1 liter.

For Nitrates. — *Standard Nitrate Solution.* — Dissolve 0.720 gram of pure recrystallized KNO_3 in 1 liter of water. Evaporate 10 c.c. of this strong solution cautiously on the water-bath, moisten quickly and thoroughly with 2 c.c. of phenol-disulphonic acid, and dilute to 1 liter for the standard solution. 1 c.c. = 0.000001 gram nitrogen.

Phenol-disulphonic Acid. — Heat together 3 grams synthetic phenol with 37 grams pure, concentrated H_2SO_4 in a boiling water-bath for six hours.

Potassium Hydroxide. — 30 per cent.

For Kjeldahl Process. — *Sulphuric Acid.* — Sp. gr. 1.84. This should be free from nitrogen. May be obtained from Baker and Adamson, Easton, Pa.

Potassium Hydroxide. — Dissolve 350 grams of the best stick potash in 2.25 liters of water and boil down to something less than a liter with 3 grams of permanganate crystals. When cold, dilute to a liter with water free from ammonia.

For Chlorine. — *Salt Solution.* — Dissolve 16.48 grams of fused NaCl in a liter of distilled water. For the standard solution dilute 100 c.c. of this strong solution to 1 liter. 1 c.c. = 0.001 gram chlorine.

Silver Nitrate. — Dissolve about 2.42 grams of $AgNO_3$ (dry crystals) in 1 liter of chlorine-free water. 1 c.c. = 0.0005 gram Cl, approximately. Standardize against the NaCl solution.

Potassium Chromate. — Dissolve 50 grams neutral K_2CrO_4 in a little distilled water. Add enough $AgNO_3$ to produce a slight red precipitate. Filter and make the filtrate up to a liter with water free from chlorine.

Milk of Alumina for Decolorization. — Dissolve 125 grams of potash or ammonia alum in a liter of distilled water. Pre-

cipitate the $Al(OH)_3$ by the cautious addition of NH_4OH. Wash the precipitate in a large jar by decantation until free from chlorine, nitrites, and ammonia.

For Hardness. — *Standard Calcium Chloride Solution.* — Dissolve 0.200 gram of pure Iceland spar in dilute HCl, taking care to avoid loss by spattering, and evaporate to dryness several times, to remove the excess of acid. Dissolve the calcium chloride thus formed in 1 liter of water.

Standard Soap Solution. — Dissolve 100 grams of the best white, dry castile soap in a liter of 80 per cent alcohol. Of this strong solution dissolve 75 to 100 c.c. in a liter of 70 per cent alcohol. This solution must have 70 per cent alcohol added to it until 14.25 c.c. of it give the required lather with 50 c.c. of the above $CaCl_2$ solution.

Erythrosine Indicator. — Dissolve 0.1 gram of erythrosine in 1 liter of water.

Methyl Orange Indicator. — Dissolve 0.1 gram Aniline Orange, Merck, (Methyl) or Orange III in a few cubic centimeters of alcohol and dilute to 100 c.c. with distilled water.

Soda Reagent. — Equal parts of sodium hydroxide and sodium carbonate solutions, the mixture to be approximately $\frac{N}{10}$.

For Iron. — *Standard Solution.* — Dissolve 0.7 gram of crystallized ferrous ammonium sulphate in 50 c.c. of distilled water and add 20 c.c. of dilute sulphuric acid. Warm the solution slightly and add potassium permanganate until the iron is completely oxidized. Dilute the solution to one liter. One cubic centimeter of the standard solution equals 0.1 mg. Fe.

Potassium Sulphocyanate. — 20 grams per liter.

Hydrochloric Acid. — One part HCl (sp. gr. 1.20) to 1 part of water.

Potassium Permanganate. — Five grams $KMnO_4$ in 1 liter of water.

For Dissolved Oxygen. — *Manganous Sulphate.* — 48 grams of $MnSO_4 . 4\,H_2O$ in 100 c.c. of water.

Alkaline Potassium Iodide. — 360 grams of NaOH and 100 grams of KI in 1 liter of water.

Hydrochloric Acid. — Sp. gr. 1.20.

Potassium Acetate. — 100 grams in 100 c.c. of water.

Sodium Thiosulphate Solution. — $\frac{N}{100}$. Dissolve 2.48 grams of the pure crystallized salt in water and dilute to one liter. Standardize against a $\frac{N}{100}$ potassium bichromate solution.

For Oxygen Consumed. — *Standard Ammonium Oxalate Solution.* — Dissolve 0.888 gram pure ammonium oxalate in 1 liter of distilled water. One cubic centimeter is equivalent to 0.0001 gram oxygen consumed.

Potassium Permanganate Solution. — Dissolve 0.4 gram potassium permanganate in 1 liter of distilled water and standardize against the ammonium oxalate solution according to the method described in the text.

For Free Carbonic Acid. — Standard $\frac{N}{22}$ Sodium Carbonate Solution.

For Lead. — *Standard Lead Solution.* — To a strong solution of lead acetate add a slight excess of H_2SO_4, filter off and wash the precipitate. Dissolve it in ammonium acetate solution, made by neutralizing glacial acetic acid with strong ammonia. Make up to a known volume and determine the lead in an aliquot part by precipitating with $K_2Cr_2O_7$ and weighing the lead chromate. Dilute an aliquot part to make a convenient standard, say about 1 c.c. = 0.001 gram of Pb.

Food Analysis

Pumice. — Bits of ignited pumice, about the size of a pea, dropped while hot into water and bottled for use.

Alcohol (for Reichert-Meissl method). — 95 per cent alcohol redistilled from potassium hydroxide.

Iodine Solution (for Hanus' method). — This is conveniently made up according to the directions of Hunt.* Dissolve 13.2 grams iodine in 1 liter of glacial acetic acid (99 per cent, show-

* *J. Soc. Chem. Ind.*, **21**, *1902*, 454.

ing no reduction with bichromate and sulphuric acid). This will best be done by adding the acetic acid in portions and heating on the water-bath with frequent shaking. To the cold solution add enough bromine to double the halogen content, as shown by titration. Three cubic centimeters of bromine is sufficient. A slight excess of iodine is not detrimental.

Anhydrous Ether. — Wash ordinary ether several times with distilled water and add solid caustic potash until most of the water has been removed. Then add small pieces of clean metallic sodium until there is no further evolution of hydrogen gas. The ether thus prepared should be kept over metallic sodium and a tube of calcium chloride should be inserted in the stopper, in order to allow the escape of any accumulated gas.

Potassium Sulphide. — Dissolve 40 grams of the crystallized salt in 1 liter of water and filter through glass wool.

Potassium Hydroxide (for Kjeldahl process). — Dissolve 700 grams of the best quality of stick potash in water and dilute to 1 liter.

Basic Lead Acetate. — Boil for half an hour 440 grams of lead acetate and 264 grams of litharge in 1500 c.c. of water. Cool and dilute to 2 liters. Allow to settle and siphon off the clear liquid. (Specific gravity about 1.27, containing about 35 per cent of the basic salt.)

Ferric Alum. — Dissolve 2 grams of ferric alum in 100 c.c. of water, boil the solution until a precipitate appears, and filter.

Fehling's Solution. — (*a*) Dissolve 69.28 grams of C.P. crystallized copper sulphate, carefully dried between blotting-paper, in water and make up to 1 liter, including 1 c.c. of strong sulphuric acid: (*b*) Dissolve 346 grams of sodium potassium tartrate and 100 grams of sodium hydroxide in water and make up to a liter.

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11. Analyses of American Feeding Stuffs. 1892.
21. Chemistry and Economy of Food. 1895.
25. Dairy Bacteriology. 1895.

28. (Rev. Ed.) Chemical Composition of American Food Materials. 1895.
29. Dietary Studies at the University of Tennessee. 1896.
31. Dietary Studies at the University of Missouri. 1896.
32. Dietary Studies at Purdue University. 1896.
34. Carbohydrates of Wheat, Maize, Flour, and Bread. 1896.
35. Food and Nutrition Investigations in New Jersey. 1896.
37. Dietary Studies at the Maine State College. 1897.
38. Dietary Studies — Food of the Negro in Alabama. 1897.
40. Dietary Studies in New Mexico. 1897.
43. Composition and Digestibility of Potatoes and Eggs. 1897.
44. Metabolism of Nitrogen and Carbon in the Human Organism. 1897.
45. A Digest of Metabolism Experiments. 1897.
46. Dietary Studies in New York City. 1898.
52. Nutrition Investigations in Pittsburgh, Pa. 1898.
53. Nutrition Investigations at the University of Tennessee. 1898.
54. Nutrition Investigations in New Mexico. 1898.
55. Dietary Studies in Chicago. 1898.
63. Experiments on the Conservation of Energy in the Human Body. 1899.
66. Creatin and Creatinin. 1899.
67. Bread and Bread Making. 1899.
69. Experiments on the Metabolism of Matter and Energy in the Human Body. 1899.
71. Dietary Studies of Negroes. 1899.
75. Dietary Studies of University Boat Crews. 1900.
84. Nutrition Investigations at the California Agr. Expt. Station. 1900.
85. Investigations on the Digestibility and Nutritive Value of Bread. 1900.
89. Effect of Muscular Work on Digestion of Food and Metabolism of Nitrogen. 1901.
91. Nutrition Investigations at the University of Illinois, etc. 1901.
98. Effect of Severe and Prolonged Muscular Work on Food Consumption Digestibility, and Metabolism. 1901.
101. Studies on Bread and Bread Making. 1901.
102. Losses in Cooking Meat. 1901.
107. Nutrition Investigations among Fruitarians and Chinese. 1901.
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126. Digestibility and Nutritive Value of Bread. 1903.
129. Dietary Studies: Boston and other Places. 1903.
132. Further Investigations among Fruitarians. 1903.
152. Dietary Studies with Harvard University Students.
162. Studies on Influence of Cooking on Nutritive Value of Meats.
227. Calcium, Magnesium and Phosphorus in Food and Nutrition.

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No. 13. Foods and Food Adulteration — (Ten Parts).
45. Analyses of Cereals.
50. Composition of Maize.
59. Composition of American Wines.
61. Pure Food Laws of Foreign Countries.
66. Fruits and Fruit Products.
69. Foods and Food Control.
72. American Wines at Paris Exposition of 1900.
77. Olive Oil and Its Substitutes.
84. Influence of Food Preservatives and Artificial Colors on Digestion and Health.
100. Some Forms of Food Adulteration and Simple Methods for their Detection.
107. Official and Provisional Methods of Analysis.
110. Chemical Analysis and Composition of American Honeys.
114. Meat Extracts and Similar Preparations.
115. Effects of Cold Storage on Eggs, Quail and Chickens.
120. Feeding Value of Cereals.
122. Annual Proceedings A. O. A. C.
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164. Graham Flour.

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No. 23. Foods: Nutritive Value and Cost. 1894.
29. Souring of Milk. 1895.
34. Meats: Composition and Cooking. 1896.
74. Milk as Food. 1898.
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112. Bread and the Principles of Bread Making. 1900.
121. Beans, Peas, and other Legumes as Food. 1900.
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142. The Nutritive and Economic Value of Food. 1901.
182. Poultry as Food.
249. Cereal Breakfast Foods.
252. Maple Sugar and Sirup.
293. Use of Fruit as Food.
332. Nuts and their Uses as Food.
363. Use of Milk as Food.
490. Bacteria in Milk.

Much valuable information will also be found in the regular bulletins and reports of several of the State experiment stations and boards of health, notably those of Connecticut, North Dakota, Maine, Kansas, New Hampshire, Vermont and Massachusetts. The "Food Inspection Divisions" and "Notices of Judgment" issued from time to time in the enforcement of the Federal Pure Food Law also contain interesting information concerning the adulteration of food.

INDEX

PAGE

Acid, sulphanilic, reagent 229
sulphuric, reagent for air analysis 228
Adams' method for fat 141
Adulteration, cause of 124
character of 128
definition of 124
extent of 128
Air, amount of, required 18
bacteria in 10, 42
collection of samples of 24
composition of expired 11
composition of inspired 9
dust in 9
essential to life 2
humidity of 9, 14
methods of analysis of 21
purification 20
poisonous gases in 10
Alcohol, determination of 191
table 217
Alkalinity, determination of, in water 92
Alum, determination of, in water 95
Alumina, milk of 230
Ammonia, albuminoid, determination of 72
significance of 61
free, determination of 72
significance of 62
free water 228
standard solution of 229
Ammonium oxalate solution, standard 232
Analysis, air, methods of 21
water, methods of 69
accuracy of methods of 59
expression of results of 58
interpretation of results of 56
Ash, in cereals 180
in wine 193
of milk 141

	Page
Babcock method for fat	142
Bacteria in air,	10
determination of	42
Barometers	21
Basic lead acetate	233
Beer, analysis of	197
Benzoic acid, detection of	196
Bibliography	234
Bleach, use of, in water sterilization	54
Boric acid, detection in milk	154
Breakfast foods	130
Butter, analysis of	165
composition of	162
Federal standard for	164
microscopic examination	178
Butter-fat, composition of	163
Calcium chloride solution, standard	231
Calorie, definition of	117
Calorific value	117
Cane-sugar, detection in milk	150
Caramel in vanilla extracts	204
Carbohydrates, function of	115
Carbon dioxide, allowable amounts in air	17
determination of, in air	27
in water	84
in expired air	11
in inspired air	9
poisonous action of	12
table of weights of cubic centimeters of	209
use as a ventilation test	17
Carbon monoxide in air	10
determination of	40
Carbonaceous matter in water, determination of	85
Casein, determination in milk	146
Cereals, analysis of	180
composition of	181
Chloride of lime, use in water sterilization	54
Chlorides in water, significance of	64
determination of	84
Chlorine, map of normal	65
in water, see chlorides.	
Cholera	44
Coal-tar dyes, detection of	194
Cohen and Appleyard method for air analysis	37
Collection of samples of air	24
water	69

PAGE
Color in water, determination of ... 105
Color, detection in milk ... 154
Colors in food ... 132
Comfort ... 11
curve of ... 16
Cooking, changes caused by ... 116
Coumarin, determination of ... 201
Crowd poisoning, theory of ... 12
Crude fibre ... 188

Dextrin, determination in cereals ... 185
Dietaries ... 120
Dust in air ... 9
determination of ... 23

Electric muffle furnace ... 88
Erythrosine indicator ... 231
Ether, anhydrous ... 233
extract in cereals ... 182
Extract in beer-wort ... 222
in wine ... 192
in wine, table ... 220

Fat, determination in milk ... 141
in cereals ... 182
Fats, function of ... 114
Fehlings' solution ... 233
Ferric alum ... 233
Ferrous ammonium sulphate, standard solution ... 89, 231
Filter galleries ... 50
Filters, water ... 52
Filtration, methods of ... 52
Fitz shaker ... 39
Food, composition of ... 112
definition and uses ... 111
essential for life ... 5
materials, composition of ... 118
principles ... 112
values, discussion of ... 122
Foods, predigested ... 120
"Fore" milk ... 138
Formaldehyde, detection in milk ... 151
Free acids, in wine ... 193

Gottlieb method for fat ... 143
Ground waters ... 48
Gunning method ... 184

PAGE
Hale and Melia method for dissolved oxygen 101
Hanus method 171
Hardness, acid method for 91
permanent, determination of 93
soap method for 90
table of 216
temporary, determination of 92
total, determination of 94
Hazen's theorem 45
Heat loss from the body, methods of 13
Heat of combustion 117
Hehner value 170
Hehner's acid method for hardness 91
Humidity 9
determination of 22
effect on heat loss of 14
table of relative 210
Hygrometer, hair 22

Ice 55
Imhoff tank 61
Indicator, erythrosine 231
methyl orange 231
potassium chromate 84, 230
Iodine value 171
Hanus solution for 232
Iron in water, determination of 89
standard solution of 231

Jackson's candle turbidimeter 95

Kjeldahl method 183
process for nitrogen in water 78
reagents for 230
Kubel's hot acid method 86

Lead in water, determination of 97
number of vanilla 202
standard solution of 232

Lemon color 207
extract 205
oil, determination of 206
Lime water reagent for air analysis 38, 228
Logwood test for alum in water 95
Loss on ignition, determination of, in water 87
Low's method for alum in water 96

PAGE
Malted cereals, analysis of ... 189
Manganese sulphate solution ... 231
Marsh test for caramel ... 204
Methyl orange indicator ... 231
Milk, composition of ... 136
detection of added water ... 158
interpretation of analysis ... 156
serum, examination of ... 149
solids, calculation of ... 148
sugar, determination of ... 145
variations in compositions of ... 137
Mills-Reincke phenomenon ... 45
Mineral matter in water ... 65
determination of ... 87
salts, value in food ... 116
Misbranding ... 125
Moisture in cereals ... 180
see Humidity.
Motion of air, determination of ... 22
effect on heat loss ... 14

Naphthylamine acetate solution ... 80, 230
Nessler reagent ... 72, 228
Nitrate solution, standard ... 230
Nitrates in water, determination of ... 82
significance of ... 63
Nitrite solution, standard ... 229
Nitrites in air, determination of ... 42
in water, determination of ... 80
significance of ... 63
Nitrogen cycle ... 60
in water, determination of total ... 78
Nitrogen-free extract ... 184
Nitrogenous substances, function of ... 113
Nutritive ratio ... 117

Odor in water, determination of ... 106
Odors in water, extermination of ... 48
Oleomargarine ... 163
Organic matter in water ... 66
determination of ... 85
Oxygen consumed, determination of ... 85
dissolved in water, determination of ... 100
significance of ... 66
in expired air ... 11
in inspired air ... 9
table of saturation of water with ... 103

	Page
Ozone, use in purifying air	20
sterilizing water	54
Pettenkofer method for carbon dioxide	33
Pettersen-Palmquist apparatus	27
Phenoldisulphonic acid reagent	82, 230
Pollution in wells, methods of tracing	50
past	63
Potassium acetate solution	232
chromate indicator	230
iodide solution, alkaline	231
permanganate, alkaline	73, 229
permanganate solution, standard	232
sulphocyanate reagent	89, 231
Preservatives, detection in butter	166
Preservatives in food	132
Protein by Kjeldahl method	182
Proteins in milk	146
Psychrometer	22
Putrescibility test	104
Radiator, platinum	88
Rain fall, average	46
water	45
Rapid methods for air analysis	35
Reagents for air analysis	228
water analysis	228
Reducing sugar, Munson and Walker's table	221
Refractive index	173
Refractometer	174
principle of	175
Reichert-Meissl number	167
Renovated butter, manufacture of	163
Residue on evaporation, determination of	87
Resins, in vanilla	203
Respiration	11
Salicylic acid, detection of	196
Salt, determination in butter	166
Sanitary science, importance of	2
Sanitation, scope of chemistry of	1
Sediment, determination of	108
Self-purification of streams	47
Septic tank	61
Sewage analysis	67, 109
purification required	44
tables of analyses of	213

PAGE
Silver nitrate solution 84, 230
Skimmed milk, detection of 161
Soap method for hardness 90
solution, standard 231
Soda reagent 93, 231
Sodium carbonate, detection in milk 154
Sodium chloride solution, standard 84, 230
thiosulphate solution, standard 232
Solids of milk 140
Sonden apparatus for air analysis 27
Specific gravity of milk 139
correction table 216
solids 162
wine 191
Spoon test 177
Standards for ammonia determination 75, 77
Starch, determination of 186
Steam vacuum method for air samples 25
"Strippings" 138
Sulphanilic acid 80, 229
Sulphates in water, determination of 95
table of 215
Sulphites, detection of 198
Sugars in cereal products 185
Surface waters 46

Temperature, determination of 21
effect on heat loss 13
Tests on water, value of 67
Thermometers, wet and dry bulb 22
Turbidimeter for sulphates 95
Turbidity, determination of 108
Turmeric in lemon extract 207
Typhoid fever 45

Ultraviolet light for water sterilization 54

Vanilla, adulteration of 200
determination of 201
extract 199
Vapor tension of water, table of 208
Ventilation 17
formulæ 18
methods of 19
Volatile acids, in wine 194

Walker method for carbon dioxide 28
Water, analytical methods 69

Page
Water, bacteriological examination of 57, 109
chemical examination of 59, 67
consumption 43
cycle of 45
daily quantity required 4
ground 48
need of 3
physiological action of 43
purification of 51
rain 45
relation to disease 44
safe 56
necessity of 45
samples, collection of 69
sanitary examination of 57
siphon method 24
sterilization of 54
storage of 46
supplies, requirements for 56
surface 46
vapor in air, *see* Humidity.
waste 43
Waters, table of average composition of 211
table of normal 212
tables of polluted 213
Wells, deep 49
shallow 49
Wine, composition of 190
Winkler method for dissolved oxygen 100

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